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100 Area Soil Washing
Bench-Scale Test Procedures

H. D. Freeman
M. A. Gerber
S. V. Mattigod
R. J. Serne

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Pacific Northwest Laboratory
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Contents

Figures

Tables

1.0 Introduction

This document describes methodologies and procedures for conducting soil washing treatability tests in accordance with the *100 Area Soil Washing Treatability Test Plan* (DOE-RL 1992, Draft A). The objective of this treatability study is to evaluate the use of physical separation systems and chemical extraction methods as a means of separating chemically and radioactively contaminated soil fractions from uncontaminated soil fractions. These data will be primarily used for determining feasibility of the individual unit operations and defining the requirements for a system, or systems, for pilot-scale testing. However, the data will not necessarily be suitable for directly designing full-scale equipment.

Besides the bench-scale test procedures, two supporting plans, a project-specific quality Assurance Project Plan (QAPjP)(Appendix B) and a Health and Safety plan (Appendix C), are included.

All work performed for this study will be conducted in accordance with the *100 Area Soil Washing Treatability Test Plan* (DOE-RL 1992, Draft A) and the QAPjP. However, the controlling document for this Soil Washing Study is the SOW (81340-92-030). The treatability tests to be performed include

- Detailed Soil Characterization
- Attrition Scrubbing
- Chemical Extraction
- Attrition Scrubbing/Chemical Extraction Optimization
- Heap Leaching
- Waste Water/Spent Extractant Treatment

The detailed procedures for conducting these tests are presented in sections 2.0 through 7.0. The schedule for conducting these tests is presented in section 8.0.

2.0 Characterization of Soils

The principal objective of soil characterization is to measure the concentrations of various contaminants in bulk and in different size fractions of the soils, and to determine the properties (chemical, physical, and mineralogical) that govern the contaminant partitioning and release behavior of soils during the washing process. Initial measurements will include chemical analyses on whole soil and particle fractions obtained by wet-sieving. The second part of soil characterization will include measurements of important properties such as moisture content, specific gravity, particle size distribution, total organic carbon, cation exchange capacity, Toxicity Characteristic Leaching Procedure (TCLP), sequential extraction, gradient density separation, optical and scanning microscopy, and X-ray diffraction analysis. The analytical levels for various tasks and analyses are listed in the QAPjP (Tables 6.1 and 6.2, Appendix B).

2.1 Chemical Characterization

The goal of this analysis is to measure the concentrations of radioactive and chemical contaminants in soil samples from 116-C-1 and 116-D-1B trenches. The chemical characterization of these soil samples will provide data to establish the levels of contamination and to identify the contaminants that exceed the specified performance levels.

Soil samples received from Westinghouse Hanford Company (WHC) will be air dried. To ensure representative soil subsamples from each trench, a method of coning and quartering (ASTM D 421-85) will be used. This is accomplished by compositing the air-dried soil into four equal batches. Next, each of these four batches will be coned and quartered and one randomly selected quarter from each of the four batches will be composited into a single batch. This single batch will be stored and representative subsamples will be drawn from this source for subsequent work. If this single-source batch of soil is used up during testing, additional source batches of soil will be prepared by compositing all the remaining soil into an appropriate number of batches and repeating the process of coning and quartering until a new single-source batch of soil is obtained.

Prior to the bench-top soil washing tests, two subsamples obtained from the composite of the entire source batch will be analyzed for concentrations of radionuclides and chromium. All analyses will be performed by the Pacific Northwest Laboratory (PNL) Analytical Laboratory (D7E15) using the documented procedures listed in Table 6.1 of the companion project QAPjP (Appendix B). We plan on analyzing two subsamples from each

trench for a total of four samples. Additional subsamples will be offered to WHC should they wish to obtain TCLP or other analyses under other subcontracts.

2.2 Wet Screening

2.2.1 Objective

The goal of this test is to find the extent to which contaminants are associated with various particle size fractions of soils. In soils, the finer size fractions, because of their larger surface areas per unit mass, usually contain larger fractions of contaminants. By preferential removal of fine fractions through wet sieving (i.e., soil washing), significant fractions of the total soil contamination can be isolated for disposal. The wet screening test will evaluate the mass distribution of contaminants within various size fractions of soil samples from the 116-C-1 and 116-D-1B trenches.

2.2.2 Method

The procedure used for wet screening will be similar to the American Society for Testing and Materials (ASTM) method D 422-63 except for the following modifications. Because the objective of this test is to examine the contaminant distribution among particle fractions, no dispersant will be used. The suggested use of a mixture of sodium hexametaphosphate and sodium hydroxide will be omitted because dispersants tend to release and redistribute the contaminants between soil and aqueous phases.

Appropriate sample quantities as specified in ASTM D 422-63 will be transferred to a sequence of sieves [25 mm, 9.5 mm, 2 mm (No. 10), 0.25 mm (No. 60), and 0.075 mm (No. 200)] and wet sieved with deionized distilled water until the wash water is clear. The soil fractions retained on the sieves will be dried in an oven at 110 $\pm 5^\circ$ C and weighed. The soil fractions will be composited to represent >2 mm, 2.00 - 0.25 mm, and 0.25 - 0.075 mm size fractions. Aliquots of soil fractions <0.075 mm will be filtered out of wash water and dried. These soil fractions and the wash water will be analyzed for the contaminants of interest using analytical methods listed in Table 6.2 in the companion QAPjP (Appendix B). Mass balance will be computed from the contaminant concentration data. The data generated are necessary to assess the contaminant mass in each of the soil size fractions and the mass released into wash water during the sieving process, a proxy for physical soil washing. We plan on performing the wet screening a sufficient number of times to yield two discrete suites of samples for contaminant analyses.

2.3 Physical, Chemical, and Mineralogical Characterization of Soils

2.3.1 Objective

These characterization studies will be used to determine the important physical, chemical and mineralogical properties of the soils. These tests include moisture content, specific gravity, particle size distribution, total organic carbon, cation exchange capacity, TCLP for metals only, sequential extraction (to elucidate binding mechanisms), gradient density separation, optical and scanning microscopy, and X-ray diffraction analysis. The information derived from these tests will be useful in analyzing and interpreting the data derived from wet sieving, chemical extraction, attrition scrubbing, and heap leaching tests. In particular, the characterization data will allow us to estimate the size fractions and the type of minerals to which the contaminants are bound. In most tests duplicate measurements will be conducted. For the more cost-intensive tests (sequential extraction, linear density gradient fractionation, microscopy, and X-ray diffraction analysis), single subsamples will be characterized.

2.3.2 Methods

Moisture Content: Gravimetric water content of the soil samples will be determined using the standard procedure (Gardner 1986). The results will be reported as the percentage of water on a dry mass basis.

Specific Gravity of Soils: The specific gravity of soil samples will be determined according to the ASTM standard test methods. For soil particle fractions larger than 4.75 mm, ASTM method C 127-88 will be used. The specific gravity of soil fraction finer than 4.75 mm will be measured by ASTM method D 854-83. The specific gravity value shall be reported as the weighted average of the two both soil fractions, as specified in ASTM D 854-83. Additionally, specific gravities of particles smaller than 2.00 mm will be determined and used in calculating particle sizes by the hydrometer method (ASTM D 422-63).

Particle Size Distribution: The particle size determination will be made according to ASTM method D 422-63. According to this method, the distribution of particle sizes larger than 2 mm (retained on No. 10 sieve) is determined by dry sieving. Soil fractions finer than 2 mm will be dispersed and the distribution of particles smaller than 0.075 mm is determined by measuring the sedimentation rate using a hydrometer. Following the hydrometer measurements, the soil sample will be washed through a 0.075-mm (No. 200) sieve, dried at 110 ± 5 °C, and dry sieved through a set of sieves (Numbers 20, 40, 60, and 140). The weight percent of soil finer than each specified size fraction will be tabulated.

Total Organic Carbon: The total organic carbon (TOC) content of the soil samples will be measured by the coulometric method (ASTM D 4129-88). In this method, soil-bound carbon is mobilized as carbon dioxide through combustion and acidification. The released carbon dioxide is absorbed into ethanolamine and measured by coulometric titration. The TOC values will be reported as percent of the mass of soil.

Cation Exchange Capacity: Cation exchange capacity of the soils will be determined according to the ammonium acetate method (Thomas 1986). Cation exchange capacity will be calculated as the milliequivalent sum of all exchangeable cations per 100 g of soil.

Toxicity Characteristic Leaching Procedure (TCLP): The TCLP was designed by the U.S. Environmental Protection Agency (EPA) to determine the mobility of both organic and inorganic analytes in wastes. The TCLP tests of untreated trench soils will be conducted according to Method 1311 (EPA 1990). The extracts will be analyzed for eight regulated metals, including Cr. The TCLP will also be performed on the coarse soil fraction obtained from the final recommended (optimized) bench-scale treatment.

Sequential Extraction: Sequential extractions of soils are conducted to gain some understanding of contaminant binding mechanisms with operationally defined groups of mineral forms in soils. The method (Belzile et al. 1989) consists of extracting soils sequentially with increasingly strong extractants; the fractions solubilized are characterized as exchangeable, carbonate-bound and adsorbed, Mn-oxide bound, Fe-Mn oxide bound, organic matter and sulfide bound, and residual mineral bound. These extraction steps are expected to provide information on specific affinities of contaminants for different types of mineral surfaces and matrices.

Linear Density Gradient Fractionation: A number of contaminants in soils exhibit both particle size and mineral specific associations. For instance, Cs is known to associate more specifically with micaceous minerals, whereas Cr(III) is known to be preferentially associated with Fe and Mn oxides and hydroxides. Density fractionation of soils contaminated with radioactivity is part of the characterization protocol (EPA 1992) because in many cases the radioactive particles tend to concentrate within heavy minerals.

The particle fractions of the trench soils will be separated into appropriate density fractions using a linear density fractionation method (Mattigod and Ervin 1983). Each of the density fractions will be characterized as to their mineralogy and contaminant concentrations by documented methods listed in Table 6.2 of the project QAPjP (Appendix B). Improved understanding of such specific contaminant-mineral associations will be useful in interpreting bench-scale test data and designing appropriate soil washing systems. For example, if a major fraction of Cs is present in the interlayers of micaceous

minerals, typical chemical soil washing and heap leaching may not be very effective in mobilizing this contaminant. Therefore it may be necessary to test various electrolytes that may release Cs from the interlayer sites.

Optical and Scanning Electron Microscopy: These microscopy techniques will be used to qualitatively determine the degree of weathering, aggregation, heterogeneity, presence of coatings, particle texture and shape, and the nature of parent material. Chemical composition of individual and aggregate particles (if needed) will be determined by energy-dispersive X-ray spectrometry. Standard techniques will be used in sample preparation, observation, data collection, and interpretation. Microscopy data on untreated and treated soil will be useful to determine any changes in the properties (aggregation, surface coatings, particle texture and shape) of soil particles after attrition scrubbing and chemical washing.

X-ray Diffraction Analysis: This technique is useful in identifying crystalline minerals, specifically minerals in the silt and clay fractions of soils. The nature and quantities of clay minerals are known to control the quantities and types of contaminants that reside in finer fractions of soils. Sand, silt, and especially the clay fractions of soils from both trenches will be analyzed using standard X-ray diffraction techniques (Whittig and Allardice 1986). Qualitative and semiquantitative estimates of the minerals present in these soils will be made. These data, in conjunction with other physical and chemical data, will be helpful in interpreting contaminant behavior during the different types of washing processes.

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3.0 Attrition Scrubbing

3.1 Objective

The objective of these tests is to determine whether contaminants can be removed from the surfaces of soil particles through scrubbing actions. These tests are not necessarily designed to provide data to directly design full-scale equipment. However, the results should show whether some form of attrition scrubbing is beneficial in partitioning contaminants to the fine fraction. The tests will evaluate a number of parameters affecting the performance of attrition scrubbing, including solids density, impeller speed, residence time and the use of surfactants, to ensure that the technique is evaluated under a set of conditions likely to result in success.

3.2 Equipment

The attrition scrubber tests will be performed in a laboratory-scale attrition scrubber fabricated from a high-torque servo-controlled stir motor with a stainless steel shaft with two three-bladed 5-cm-diameter impellers (Figure 3-1). The impellers will be placed on the end of the steel shaft with the blades facing with opposing pitch. This configuration maximizes the particle-to-particle contact that results in the desired scrubbing action. The motor speed is continuously adjustable from 50 to 900 rpm and the speed is maintained by a servo control loop to ensure reproducibility between tests. The motor controller also has a built-in timer to allow the contact times to be controlled precisely. The mix container can be almost any size, although a 1-liter stainless steel square or cylindrical container will likely be used.

3.3 Procedures

Depending on the texture of the soil material, two different approaches are necessary to evaluate the effectiveness of attrition scrubbing in contaminant removal. Attrition scrubbing for <2mm fraction is evaluated by conducting stirred agitation tests over a range

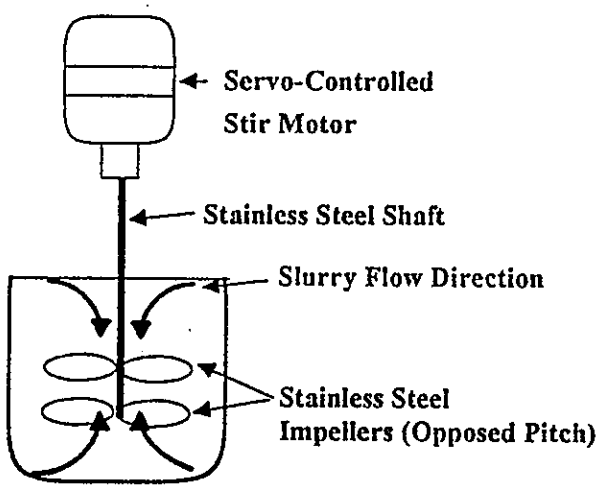

Figure 3-1. Laboratory-Scale Attrition Scrubber

of conditions including solids density, impeller speed, contact time, and surfactant addition. These tests are performed at room temperature using the following procedure:

1. Weigh 500 ± 0.1 grams of dried soil and place in the attrition scrubber container.
2. Add the appropriate amount of deionized water or surfactant solution (5-10% by weight) surfactant solution and mix.
3. Program the mixer controller for the appropriate time and speed.
4. Start the stir motor and slowly lower into the scrubber container.
5. Allow the slurry to mix until the timer stops the motor rotation.
6. Wet screen the slurry through a suitably fine [e.g., 200 mesh (75-μm)] screen using deionized water to wash the fines through the screen. Collect all of the fines for further processing.
7. Filter the fines through 0.45-μm filter paper.
8. Dry both fractions (filtered fines and coarse gangue) in an oven at 60°C for 24 hr.
9. Weigh each fraction on an analytical balance to the nearest 0.1 gram.

10. Submit a representative portion of coarse and fine fractions for gamma and beta counting and metals analysis, if appropriate. A selected number of tests (about 10%) will include the analysis of the liquid portion by gamma and beta spectroscopy to obtain material balance for the contaminants if necessary (i.e. analyses of solids after attrition yield <85% recovery of original mass of contaminant).

For soil materials containing a large fraction of cobbles, the field-scale soil washing is conducted with a combination of high pressure sprays and scrubbing in the trommel. Due to large amount of cobble material in samples from 116-C-1 trench, the most likely approach will include high pressure sprays or autogenous grinding(no steel balls are added to the mill) in ball mills. The high pressure sprays should work if the contaminant is present as adhering particles or weakly adsorbed on the surface. However, if the contaminant is present in micropores or strongly adsorbed some form of grinding will be required. The following steps are proposed for decontaminating the cobbly materials.

1. Hand scrub the contaminated cobbles with stiff brush and water to determine the ease of removal of contamination.

2. If step 1 does not provide satisfactory results, use mild chemical such as dilute HCl, or citric acid with hand scrubbing.

3. If steps 1 or 2 are successful, proceed to test high pressure sprays to compare the effectiveness with hand scrubbing. If steps 1 or 2 are not successful it is unlikely that high pressure sprays will work.

4. If steps 1 and 2 are not successful, use a laboratory-scale ball mill to investigate the effects of contact time and amount of fines and sand present in the mixture upon removal of contaminants from the surfaces of cobbles. Due to the apparent small amount of <2mm material in the 116-C-1 samples, if necessary additional sand will be added to the ball mill to improve the grinding efficiency.(Note: Soils of finer texture were obtained from the first test pits. These material could be used). Because the cobbly material contains the highest levels of radioactivity, adding sand the mix during ball milling will reduce the radioactivity levels of the soil material as a whole).

5. Ball mill with dilute reagents as in step 4.

6. If steps 1-5 are not successful then proceed to the chemical extraction tests, modified for use with large cobbles.

The primary parameters affecting attrition scrubbing of <2mm soil particles are pulp density, agitator speed, residence time, and chemical addition. A limited range of each of these factors will be evaluated for the feasibility tests. A test matrix reflecting which tests will be conducted is shown in Table 3-1. This table is an estimate of the parameters to be varied based on expected soil properties and may change if the soil characteristics are significantly different than anticipated. The two surfactants that will be used at concentrations of 5 and 10% by weight will be selected based on further literature review. Initial scoping tests may be conducted to determine the best solids density and impeller speed to use as the baseline case. Sediment fractions from the attrition scrubbing tests will be analyzed for particle size and gamma activities. Selected samples will also be analyzed for alpha, beta, and metals if these types of contaminants are shown by the initial characterization studies to be present at concentrations above the action levels.

Table 3-1. Experimental Design of Attrition Scrubbing Tests for <2mm Soil Material

	RPM=900[1]								**Solids=75%**							
	60% solids				75% solids				900 rpm				450 rpm			
Time, minutes	1	5	10	15	1	5	10	15	1	5	10	15	1	5	10	15
Trench 116-C-1																
without Surfactant	x	x	x	x	x	x	x	x		x		x		x		x
with Surfactant 1		x		x		x		x		x		x		x		x
with Surfactant 2		x		x		x		x		x		x		x		x
Trench 116-D-1B																
without Surfactant	x	x	x	x	x	x	x	x		x		x		x		x
with Surfactant 1		x		x		x		x		x		x		x		x
with Surfactant 2		x		x		x		x		x		x		x		x

1. The test parameters in this table are based on preliminary judgments and are subject to change based on the characteristics of the soil samples and results of preliminary scoping tests. Optimum performance is obtained by maximizing pulp density, minimizing time of scrubbing for a maximum removal of contaminants from sand fractions.

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4.0 Chemical Extraction

4.1 Objective

The objective of the chemical extraction task is to evaluate the feasibility of removing contaminants from 100 Area soils using chemical solutions. This operation may be used if physical separation techniques do not remove contaminants to required levels or as a replacement to soil separation in the event that chemical extraction of the entire soil volume is more economical.

4.2 Background

The chemical extraction of solids to selectively remove elements of interest is a proven technique used in the metallurgical and chemical processing industries for many years. The success of this technique generally lies in the proper selection of extractants (chemicals) and in understanding the kinetics of the reactions of concern. With this information, the proper selection of equipment to perform the extraction can be made and further scale-up studies can be conducted.

For the purpose of processing large quantities of soils, three main processing methods are available. The first choice is to use a stirred vat where contact of the leachate and soil can be easily controlled. The equipment for this process is relatively simple and can be scaled to handle very large volumes. The process can be operated in a continuous mode if a number of vats are used in series, or operated in a batch mode with a single vat. The second possibility for leaching the soil is to add the leachate at the start of the soil washing process (e.g., in the trommel) and allow the reactions to take place while the physical separation is being performed. The advantage of this method is that very little equipment besides the soil washing system is needed. However, the solids-to-liquid ratios and contact time may not be optimal for the extraction to be effective. The third method is to use heap leaching, in which soil is piled on an impermeable pad and sprinkled with leachate solution. The solution percolates through the soil pile and is recovered from the underlying pad. This technique will be the primary focus of another task in this study.

4.3 Chemical Selection

A preliminary review of the literature was performed to identify candidate reagents for leaching soil to remove cobalt, europium, chromium, strontium and cesium. Table 4-1 summarizes those reagents that were identified as possible candidates for one or more of the constituents of concern. Prior data (100 Area Soil Washing Treatability Test Plan DOE/RL-51 Draft A) indicate that Pu, tritium and U activities are significantly below the specified performance levels, therefore although these are "contaminants of concern"(in 100 area work plans) they are not included for this test.

Mineral acid dissolution has a potential for treating soils containing chromium and europium. Dilute HCl is used in the mineral processing industry to leach rare earths, including europium, from ore (Considine 1974). Experiments at Rocky Flats indicated that 2N HCl was efficient in leaching plutonium and americium from Hanford soil (Stevens et al. 1982). The performance for americium may be relevant because it is immediately below europium on the periodic chart.

Table 4-1. Candidate Chemicals for Chemical Extraction Tests

Reagent	Cobalt	Europium, Chromium	Strontium	Cesium
HCl		X		
Formic Acid				X
KCl				X
NH_4CO_3				X
EDTA	X	X	X	
DTPA	X	X	X	
NTA	X	X	X	
Acetic Acid			X	X
Citric Acid	X	X		
Palmitic Acid			X	X
Stearic Acid			X	X
d-Glucuronic Acid			X	

Organic acids may be used for dissolution of cesium. Formic acid was used successfully by Bray et al. (1984) to elute cesium from ion exchange columns.

Ion exchange using salts may also be applicable to the removal of cesium and strontium from the soil (Dragun 1988). According to Dragun, the typical order of alkali adsorption on soil is Cs > Rb > K > Na > Li. Gee et al. (1983) showed that sodium chloride in solution would reduce the adsorption of cesium on montmorillonite with a K_d reduced from over 1000 mL/g at 0.002M NaCl to less than 20 mL/g at a 1M NaCl concentration. Potassium and ammonium salts compete even better than sodium salts. For alkaline earths, the order is Ba > Sr > Ca > Mg. If strontium is coprecipitated in the soil with calcium on apatite ($CaPO_4$) or carbonate ($CaCO_3$), it may be too tightly bound for ion exchange to be practical. Nelson and Mercer (1963) reported using 2M ammonium carbonate to elute cesium from ion exchange materials including clinoptilolite. 2M ammonium chloride was used as an eluting agent for removing tightly bound cesium from zirconium phosphate, and 2M ammonium chloride in 0.1M HCl to elute cesium from ferrocyanide molybdate (Baetslé et al. 1964).

Several complexants may have potential for complexing cobalt, europium, chromium, and strontium. Research conducted by PNL on cobalt mobility in soil indicates that EDTA and DTPA would be good candidates for cobalt and DTPA would be a good candidate for europium (Swanson 1983). Research by Huang et al. (1985) on adsorption of complexed cobalt on activated carbon further supports EDTA as a candidate. Review of a text on complexant stability constants by L. G. Sillén (1971) suggests that EDTA, DTPA, and NTA would be applicable to varying degrees for cobalt, europium, chromium (III), and strontium. Acetic acid was also indicated as a possible complexant for all four constituents, but only in concentrated form. This may require either lower performance in dilute solution (also indicated) or recovery of the acetic acid. According to data in Sillén's text, NTA may be a good candidate for europium, cobalt and strontium. NTA is relatively nontoxic (Windholz 1976) and readily biodegrades (Lyman et al. 1982). EDTA, on the other hand is relatively nonbiodegradable. The Draft RI/FS Work Plan for the 100-BC-1 operable unit indicated that oxalic acid and citric acid were periodically added to the ponds servicing the trench and thus could be responsible for mobilizing the contaminants. Both appear to have moderate complexing behavior for europium, cobalt and strontium (Sillén 1971). Two surfactants were identified that may be relevant to leaching cesium and strontium. Dragun (1988) referenced research by Toste et al. (1984) implicating palmitic and stearic acids as ligands that complexed cesium and strontium in the soil. Glucuronic acid has been found effective in removing Sr-90 from mineral soils (Francis, 1978).

The most likely candidates for the chemical leaching tests are HCl, acetic acid, EDTA, NTA, and KCl. Citric acid could be a candidate from a historical perspective, because it is classed as a detergent builder, as are EDTA and NTA.

4.4 Equipment

The leach tests will be performed using a Phipps & Bird Model 7790-400 Laboratory Stirrer containing six stirring paddles, all running at the same speed. The stirrer is capable of using up to 1-L beakers. In tests where temperature control of the solution is necessary, the top portion of the stirrer will be mounted over a temperature-controlled water bath containing the beakers. This same equipment may also be utilized in performing flocculation tests for the waste water treatment studies.

4.5 Procedures

The procedures for equilibrium and kinetics tests are basically the same. Tests will be conducted by contacting leach solutions with soil, stirring for a set time, then filtering and analyzing the solids and liquids for the contaminants of concern. The parameters that will be investigated in this task are leachate composition (including pH and Eh), contact time, and temperature. Up to six different chemicals will be tested at room temperature. The chosen concentrations will be within the range of 0.5-3M for HCl, KCl, and ammonium carbonate, and 0.05-0.2 M for all other extractants. Additional tests at other temperatures will be conducted on the two most promising extractants.

Parameters to be monitored frequently during the tests are temperature, pH, and Eh. Temperature will be measured periodically with thermocouples, and pH and Eh will be measured according to PNL-approved technical procedures (Appendix A). The specific procedure to be followed for these test is described below.

1. Weigh 100 g ± 0.1 g of soil and place in 500-mL beaker(s).

2. Position beakers under stir paddles on laboratory stirrer.

3. Lower paddles into beakers, leaving ~1 cm clearance on bottom of beaker.

4. Add 300 mL of leach solution to each beaker.

5. Set stirrer control to desired impeller speed.

6. Measure solution temperature, pH, and Eh.

7. Turn on stirrer and allow to stir for desired time period.

8. Turn off stirrer and lift stir paddles above top of beakers and allow slurry to drain into beaker.

9. Wash residual solids off of stir paddles with a small amount of deionized water.

10. Filter each beaker through a separate 0.45-μm filter.

11. Measure and record filtrate volume.

12. Analyze filtrate for contaminants of concern and store excess for further analysis.

13. Weigh filter cake and record results.

14. Dry filter cake in oven at 105 °C for 24 hr.

15. Weigh dried filter cake and record results.

16. Analyze filter cake for contaminants of concern using PNL procedure specified in Table 6.2 of the QAPjP (Appendix B).

Equilibrium leach tests will contact the soil and leach solution for minimum of 4 hr. and the leachate will only be sampled at the end of the test. For time-series studies, a 15-mL aliquot of slurry will be periodically removed from each beaker, filtered and analyzed according to the test procedure. This approach assumes that there is sufficient contamination in the soil to result in measurable contaminant levels in 10 mL of leachate. Duplicate tests will be conducted in accordance with the QAPjP. It is anticipated that a minimum of 48 chemical extraction tests will be performed in this task.

5.0 Optimization

The objective of this task is to determine the optimum combination of attrition scrubbing and chemical extraction test conditions with the goal of achieving 80 - 90% volume reduction of the contaminated soil. This will be achieved by investigating the combination of attrition scrubbing and chemical extraction and obtaining more information on the reaction kinetics for promising extractants. Data from this task will provide useful information for defining the operating parameters of a pilot-scale system. The specific parameters that will be investigated as part of this task will depend on the results of the previous attrition scrubbing and chemical extraction tests. The types of tests that may be performed include

1. Combining chemical extraction with attrition scrubbing.

2. Performing sequential leaches of the solids with different chemical extractants.

3. Investigating the effects of temperature on extraction rate to determine if the process can perform under winter conditions.

4. Performing detailed kinetics studies on the most promising chemical extractants so performance of the leach system can be estimated for different residence times.

The total number of tests to be conducted in this task will range from 24 to 48, depending on the number of parameters that must be optimized during this phase. The optimum parameters found in this task will be confirmed by replicating the tests with the optimum parameters. Tests to be performed under this task will follow the procedures outlined in sections 3.0 and 4.0 of this document, with minor modifications.

6.0 Heap Leaching

Heap leaching is a technique commonly used in the mining industry to recover metals from low-grade ores and tailings piles. The technique entails placing coarsely crushed ore or tailings on an impermeable pad such as clay or asphalt. A leach solution is distributed over the surface of the pile using standard sprinkler systems. The leach solution percolates through the ore pile and the metal-bearing solution is recovered from the impermeable pad for further processing. The main advantage of this system is the relatively low capital and processing costs required for successful operation.

The factors involved in a successful heap leaching operation at Hanford include the permeability and homogeneity of the soil, kinetics of the chemical extraction processes, and the ability to control fugitive dust from the piles. Tests in this task will focus on the first two factors, permeability and chemical kinetics.

The feasibility of heap leaching will be determined primarily with the use of saturated column leach tests. These tests will pass chemical extractants through a column of contaminated soil. The degree of extraction versus total volume of extractant passing through the column will be determined for each column. The tests will be performed in accordance with PNL procedure G-O I-SC (Appendix A). For some tests, the permeability of the soil before and after leaching will be measured to determine whether reactions within the pile cause plugging and uneven flow paths for the chemical extractant.

A maximum of 16 column tests will be conducted under this task. This will allow duplicate samples to be conducted for two soil types with two different chemical extractants per soil at two different flow rates. The specific chemicals and tests conditions will be determined based on the results of the chemical extraction tests.

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7.0 Waste Water/Chemical Extractant Treatment

7.1 Objective

The objective of these tests is to determine if contaminants and fine particulates can be removed from the solutions generated during soil washing and chemical extraction. These tests are not necessarily designed to provide data to directly design a full-scale waste water treatment system. However, the results should provide an indication of the potential performance of conventional waste water treatment unit operations in treating these waste streams. Waste water treatability tests will separately evaluate soil washing solutions from the attrition scrubber and the leachate from the more promising chemical extraction tests. Treatment of both suspended solids and dissolved contaminants will be considered. Tests conducted for each class of waste water are described below.

7.2 Soil Washing Water

Most of the contamination of the soil washing water is expected to be in the form of fine suspended particulate material. However, a portion of the contamination may be in the form of dissolved solids. Characterization of the waste water will be conducted to assess the degree of contamination by each mechanism. Waste water samples will be obtained from the wash and rinse steps in the soil washing experiments and filtered through a 0.45-μm filter, and the filtrate and filter cake will be analyzed for contamination and the filter cake weighed. The filtrate will then be centrifuged to remove any remaining submicron particulates and both filtrate and centrifuged solids will be analyzed. In addition, the filtrate will be analyzed for other dissolved species such as Na, K, Ca, Mg, Cl, and CO_3 to better understand the chemistry of wash water. Such data will be useful for calculating the consumption and costs of treatment reagents and ion exchange resins. Using these results, waste water solutions will be prepared that simulate the composition of wash water with respect to dissolved solids that would be present if the solution was recycled prior to treatment. The goal of the latter tests is to gather data on "worst case" wash waters that contain a heavy burden of contaminants resulting from several cycles of washing.

Treatment of suspended solids will focus on sedimentation and filtration. Waste water samples will be treated with selected flocculating/coagulating reagents over a range of concentrations. Sedimentation and filtration tests will be conducted on the simulated waste water to evaluate the performance of selected coagulating and flocculating reagents. The

reagents to be tested will be based on the chemical analysis of the fine fraction of the soil following attrition scrubbing. Both pH and Eh will be monitored during the mixing of reagents. Following solids separation, the wastewater will be analyzed for contaminant concentration to assess performance and the volume of filtrate will be measured.

Treatment of dissolved solids will focus on cation exchange materials. The specific materials evaluated will depend to some extent on the contaminants that are dissolved in solution, but may include ion exchange resins, zeolites, and activated charcoal. Batch contact tests will be conducted with each material tested to determine the distribution coefficient and capacity of the material for the contaminants of interest.

7.3 Spent Chemical Extractant

Most of the contamination of the leachate waste water is expected to be in the form of dissolved or complexed material. However, a portion of the contamination may be associated with the suspended particulate in the leachate. Characterization of each leachate of interest will be conducted in the same manner as was done for the soil washing wastewater tests, except that the filtrate will be also be analyzed for extractant concentrations. Using these results, waste water solutions will be prepared that approximate a composition of leachate with respect to dissolved solids that would be produced depending on whether leachate recycling prior to treatment is desired. This will be determined following the chemical leaching tests.

Treatment of suspended solids and dissolved solids will generally follow the same approach as described for soil washing waste water. However, the presence of complexants introduces the possibility that cation exchange will not be effective. Therefore additional tests will be conducted as necessary to identify alternative means of separation. It is anticipated that chemical oxidation using chlorine or hydrogen peroxide to destroy the complexant, ion exchange using anion exchange resins, and coagulation/ flocculation using cationic polyelectrolytes will be tested.

8.0 Schedule

The projected schedule for accomplishing the tasks outlined in this document is shown in Figure 8-1.

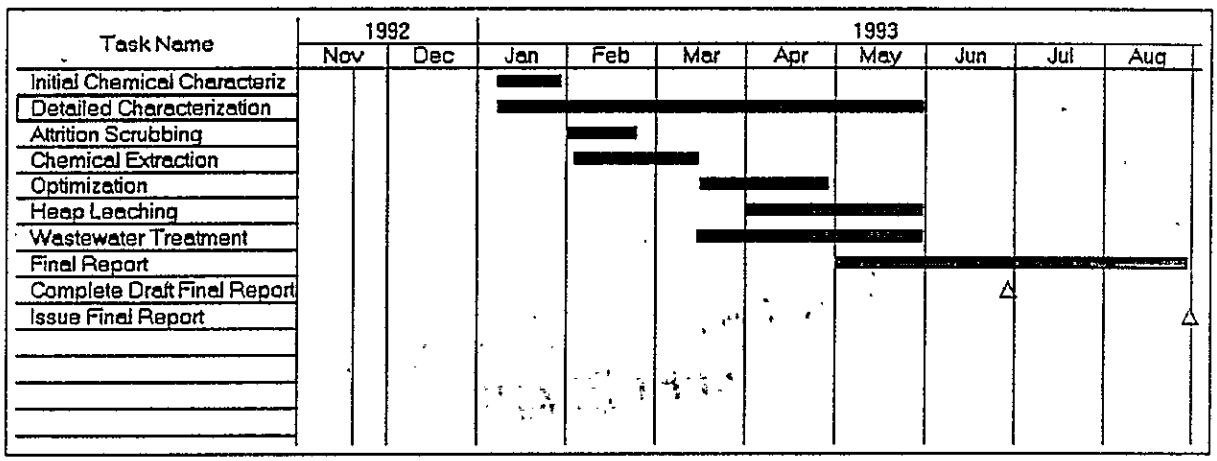

Figure 8-1. Schedule for 100 Area Bench-Scale Treatability Study

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Appendix A

PNL Technical Procedures

TITLE: G-01-SC: PROCEDURE FOR CONDUCTING SATURATED COLUMN EXPERIMENTS

1.0 APPLICABILITY

The procedure describes the method for performing saturated flow-through column experiments to study selected component(s) migration through and/or away from a solid material (i.e. sediment, waste form, etc.).

This procedure applies to all saturated column leaching experiments conducted in support of the Hanford Grout Technology Program.

2.0 DEFINITION

2.1 Column Experiment - an experimental system which continually passes a synthetic or untreated solution through and/or onto the solid material for the purpose of understanding leaching characteristics and selected component(s) migration.

2.2 Synthetic Solution - the use of laboratory grade chemicals in deionized water to reproduce a groundwater composition or enhance the concentration of a particular species of interest in solution.

2.3 Untreated Solution - the natural ground water, river water, process water, etc. The use of a solution collected as is or filtered.

2.4 Solid Material - the substance placed in the column for studying the leaching aspects and/or the migration or movement of selected component(s).

3.0 RESPONSIBILITIES STAFF

- Task Leader
- Cognizant Staff

4.0 PROCEDURE

4.1 MATERIALS

Solid Material (geologic media, crushed or intact waste form, etc.)
Column Apparatus (see Figure 1 and para. 4.5.1)
Pump (syringe, peristaltic, etc.)
Fraction Collector (if necessary)
Graduate Cylinder

QAO Approval 3-31-87	Approved (Line Manager) 4-6-87
Prepared by Wayne J. Martin	Technical Review

Procedure No.	Revision No.	Date Issued	Page
G-01-SC	0	4-15-87	1 of 13

Contact Solution
Burette
Balance (accuracy of ± 0.01 mg)
Radionuclide Tracer (if necessary)
Clamps
Tubing
Syringes
Culture Tubes
Counting System
Teflon Tape
Liquid Scintillation Vials
Insta-gel (Scintillation Cocktail)
Pipette
Nitrogen Gas
Container for Liquid Storage
Pasteur Pipette
Mortar and Pestle

4.2 SAFETY PRECAUTIONS

4.2.1 Chemicals - Special attention should be given to appropriate handling instructions for solid or liquid chemicals before proceeding with work.

4.2.2 Radionuclides - If radionuclides are to be used in column work, a Radiation Work Procedure (RWP) must be obtained before experimentation can begin. To help reduce the possibility of contamination to the laboratory or yourself rubber gloves, safety glasses and lab coat should be worn while handling radioactive solutions.

4.3 SAMPLE PREPARATION

4.3.1 The solid material shall be used as is or crushed with an apparatus that will not add particulates to the sample which could change the composition of the sample. For example, by crushing with a metal crusher, pieces of metal could be added and may cause reactions not related to the solid material under investigation (such as $Fe^{2+} \rightleftharpoons Fe^{3+} + e^-$). The foreign material could reduce, precipitate, or complex other components involved and cause an interference with selected component(s) migration through or away from the solid material. We suggest crushing sample with a diamonite mortar and pestle. Diamonite is an extremely dense and hard material which should not break off while crushing. Document the sample preparation method in the laboratory record book.

4.3.2 The solid material should be well-mixed to ensure uniform distribution of particle size throughout the column. If needed, the particle size can be determined using procedure G-03-PS. Another parameter which may be of interest is the amount of available surface area. This parameter gives an indication of reactive area available for interaction with the influent solution and can be used for other calculation purposes. If needed, the surface area can be determined for crushed or granulated solid material using procedure G-03-SA. For intact solid material the geometric surface area can be calculated from it physical dimensions.

4.3.3 Allow the sample to air dry and determine the moisture content as described in procedure G-04-MC. Record this information on the data sheet.

4.4 LOADING THE COLUMN

4.4.1 Determine the column volume either volumetrically by adding a measured amount of solution or calculate from its physical shape (i.e. for a cylinder ((πr^2) x length)). Record this information on the data sheet. Refer to paragraph 4.10.1 for instructions on column size and dimensions.

4.4.2 For intact solid material that doesn't fill the available void space, the sample should be suspended in the column with string, on glass beads or some material which will not contaminate the influent solution or the solid material. Once the intact solid material is in place and the column is sealed proceed to paragraph 4.5.

4.4.3 For granulated solid material, estimate a desired bulk density (equation 1) and weigh out the necessary amount (accounting for moisture). Then place a small portion e.g. 10-15 grams (this amount is arbitrary) of solid material into the column.

4.4.4 Tamp the solid material firmly into the column. (Suggestion: The object used for tamping should have a diameter slightly smaller than that of the column. This will ensure even packing.)

4.4.5 Then add another portion and repeat steps 4.4.3 and 4.4.4 until the column is full. If the pre-measured amount of solid material was adequate then the bulk density is known, if not, the amount not used or added must be accounted for and the bulk density recalculated (equation 1). This value should be accurate out to the 0.01 grams. Record the ID. number of the balance used on the data sheet.

Equation 1

$$\text{Bulk Density} = \frac{\text{Solid Material (g)}}{\text{Column Volume (cm}^3\text{)}}$$

4.5 INFLUENT SOLUTION

4.5.1 If the solution needs to be altered or changed it will be considered a synthetic solution. Therefore, the chemical additives used should be carefully measured with a pre-calibrated balance to ensure an accurate chemical composition and the balance number recorded on the data sheet. If reproducing a groundwater composition, then the chemicals should be prepared in de-ionized water.

4.5.2 For solutions that are to be used in a reduced condition (low oxygen content), the solution should be bubbled with nitrogen gas (or an inert gas) overnight to reduce its oxygen content. This helps reduce the possibility of oxidizing the solid material once the experiment begins. The solution storage container should be sealed from the atmosphere. Nitrogen purging should be repeated periodically to help keep the solution oxygen content low. If the environmental aspects of the experiment call for extended reducing conditions then column experiments should be performed in an atmospheric chamber where the oxygen content can be controlled.

4.5.3 All solutions shall be filtered through a 0.45 μm filter before use. This will remove any suspended particulates or undissolved chemical that might be in solution. Container used for solution storage should be leached with 0.1 N HCl and rinsed several times with distilled water before use.

4.5.4 All solutions should be characterized by having its chemical composition determined by ICP (inductively coupled plasma), GFAA (Graphite Furnace Atomic Absorption), pH, redox potential, alkalinity, IC (Ion Chromatography) and TC (total carbon). These tests will give an absolute characterization of either natural ground water or synthetic solution. Element concentration (ppm) will be reported from these tests and recorded in the laboratory record book. These data shall be converted to milliequivalents so that the cation-anion charge balance can be checked.

4.6 RADIONUCLIDE SPIKE PREPARATION (If radionuclides <u>are not</u> needed in your column experiment then proceed to section 4.7.)

4.6.1 In the following section the term original stock solution refers to a sample of concentrated isotopic solution from which dilutions are made. <u>Handle solution with care</u>.

The spiked solution refers to the diluted solution prepared from the original stock solution and is usually at a low concentration for experimental use. The same precautions should be taken as handling the original stock solution.

All samples in this example are prepared in 15 mL of solution. This amount should be determined by your radiocounting geometry which can be explained by the radiocounting analyst. The geometry is related to the standard, which is National Bureau of Standards (NBS) traceable, used in calibrating the detector.

4.6.2 Take a small aliquot of the original stock solution and count with the appropriate detection system for:

a. gamma-emitter, place 10λ of stock in 15 mL of synthetic solution.

b. alpha or beta emitters, place 10λ of stock in 15 mL of insta-gel cocktail for counting by liquid scintillation.

4.6.3 From the counts per minute (cpm) data, you can determine an appropriate dilution factor for preparing the spiking stock solution concentration. You should strive to get about 2000 to 20000 cpm/mL in the spike solution.

4.6.4 All cpm data shall be converted into disintegrations per minute (dpm) and used in calculating radionuclide concentration (refer to section 4.10.8). These results will be recorded in the laboratory record book.

4.6.5 All effluent solution samples should be placed in the predetermined sample geometry during the subsampling for the various solution analyses to be performed.

4.7 FLOW RATE DETERMINATION

4.7.1 If you're using a graduated cylinder for sample collection proceed to Section 4.7.6.

4.7.2 If a fraction collector is your effluent sampling apparatus proceed to Section 4.7.3.

4.7.3 Fill the fraction collector tube racks with culture tubes. Tube size is optional although the longer the tube, the smaller the amount of evaporation on long runs.

4.7.4 Mark, weigh, and record, in the laboratory record book, each tube weight in the order in which they will receive effluent samples. Place them in that same order in the fraction collector.

4.7.5 After the run, remove the tubes in order, weigh and record the tube plus effluent sample weights. If long runs are required (more than one day), remove the filled tubes each day and determine effluent weights. These results will be used to calculate flow rate (see Section 4.10.7). The tubes are now ready to be subsampled for characterization. Record the data from this step in the laboratory record book.

4.7.6 If using a graduated cylinder for effluent sample collection record the effluent amount and the change in time. This data gives a mL/min flow rate. Take several readings to determine an average flow rate.

4.8 SUBSAMPLING

4.8.1 The effluent samples shall have pH and Eh (redox potential) measurements taken as outlined in procedures G-05-PH and G-06-EH, respectively.

4.8.2 All effluent samples will be filtered through 0.45 μm filters and subdivided for solution analysis. The number and types of analysis is directly related to what component(s) in solution are of interest. These analyses should be similar to those performed on the influent solution.

4.9 COLUMN EXPERIMENT TERMINATION

This time factor is arbitrary and can be predetermined by several different methods such as;

- The amount of influent solution available.
- Influent and effluent concentrations of component(s) interest become equal.
- Color change of solid material.
- Flow rate change.
- Or any reason for which your purpose for conducting the experiment has been reached.

4.10 CALCULATIONS

4.10.1 Column Dimensions

The basic column should have a packing length of at least 4x or 3x that of the inside diameter of the column. This will ensure that the influent solution will contact a greater percentage of the solid material. The column should be constructed from a transparent material, so you can visually examine the solid material. The column material should be inert to the influent solution or the solid material.

4.10.2 Percent Moisture Content (refer to procedure G-04-MC for determination technique)

$$\frac{\text{(Soil Air Dry Weight - Soil Oven Dry Weight)}}{\text{Soil Oven Dry Weight}} \times 100$$

4.10.3 Bulk Density

$$\frac{\text{Oven Dry Weight of the Solid Material (g)}}{\text{Volume of the Column } (cm^3)}$$

4.10.4 Particle Density

Refer to reference by Black (1965) the procedure for determining the particle density. The formula used in the aforementioned book:

$$DP = \frac{dw(Ws - Wa)}{(Ws - Wa) - (Wsw - Ww)}$$

where

dw = density of water in grams per cubic centimeter at the temperature observed,

Ws = weight of pycnometer plus soil sample corrected to oven-dry condition,

Wa = weight of pycnometer filled with air,

Wsw = weight of pycnometer filled with soil and water, and

Ww = weight of pycnometer filled with water at the temperature observed.

4.10.5 Porosity

$$1 - \frac{\text{Bulk Density}}{\text{Particle Density}} \text{ or } \frac{\text{Pore Volume}}{\text{Column Volume}} = \text{porosity}$$

where

Column volume = cross sectional area of column x length of column

4.10.6 Pore Volume

- Volumetric Determination; First weigh the empty column, then after column is packed with dry solid material, weigh the entire column. Then use a burette to fill the column with influent solution (Figure 2).

- Determine the number of mLs it takes to fill the column. Then weigh the column again and record these values in the laboratory record book.

- pore volume = wt of column after filling
  - wt of column before filling.

- Pore volume can be calculated from previous data collected. (Remember 1 mL = 1g)

  Calculated Pore volume = (Porosity) (Column Volume)

- Compare the calculated value with the volumetric value, in some cases the calculated value will be greater then the volumetric. This is probably due to trapped air bubbles within the pores spaces within the column. The volumetric value is regarded in the literature as the effective pore volume.

4.10.7 Flow Rate Calculation

- From the procedure section Flow Rate Determination, take the difference in weight of the tubes empty and with effluent, get an average weight per sample with a standard deviation.

- To determine flow rate:

$$\frac{\text{average sample weight (mL)}}{\text{amount of time per sample (min)}}$$

- The amount of time per sample is obtained from the fraction collector setting. This is how much time the tube was allowed to sit before changing to the next tube. Flow rate is now in units of mL/min. Convert from mL/min to m/yr as follows:

- Determine the amount of time it takes the solution to traverse the column.

- $\frac{\text{pore volume (mL)}}{\text{flow rate (mL/min)}} = \text{min (A)}$

- $\frac{\text{length of column (cm)}}{\text{A (min)}} \times 5.256 \times 10^3 \text{ m/yr}$

4.10.8 Determining Disintegrations per Minute (dpm)

$$dpm = \frac{cpm}{\varepsilon\mu}$$

where

cpm = counts per minute

ε = efficiency of the detector

μ = absolute abundance of nuclide

4.10.9 Hydraulic Conductivity

Method for determining this value is referenced from Method of Soil Analysis (refer Black et. al 1965).

Here is the formula used:

$$K = \frac{(\Delta Q)/(L)}{(\Delta t)\ (A)\ (\Delta H)}$$

where

ΔQ = amount of liquid collected (mL)

L = length column (cm)

Δt = change in time (min)

ΔH = change in Head (cm)

A = cross-sectional area of column (cm^2)

4.10.10 Calculations for Plotting the Breakthrough Curve for a Specific Component

This plot involves several values from the experiment, all of which are explained separately in the following steps. Table 1 shows an example of how to organize the data for plotting purposes.

1. Sample number - the number of each culture tube in order of which it was run through the fraction collector.

2. Change in sample volume (Δ mL) - this number is calculated from individually adding the amount of effluent which was collected in each culture tube. This number accumulates with increasing sample number. The final value should represent the amount of solution which passed through the column.

3. Counts per minute (cpm/mL) - the activity detected for that sample divided by ΔmL.

4. Disintegration per minute (dpm/mL) - using the cpm/mL refer to Determining Disintegration per minute (dpm) section 4.5.8.

5. Actual dpm per sample (dpm) - The dpm/mL x ΔmL.

6. Adjusted change in volume (mL) - this is the halfway point between sample volumes. This milliliter value is used for plotting because the counts eluted for any particular sample are actually an average for that sample. Therefore the x axis plotting point will be the average of the change in volume. The following equation can be used in determining this point.

$$\text{Adj. Vol. (mL)} = \sum_{n=1}^{i=1} \Delta mL_{i-1} + \frac{1}{2} \Delta mL_i$$

7. Dpm of effluent/Dpm of influent (Ce/Ci) - the Ci value is predetermined from your influent solution. For this example the Ci = 1.0e-2 μCi/mL. The dpm value can be converted to curies using the conversion factor of 2.22 dpm/pCi. The literature states that breakthrough occurs at 0.5 Ce/Ci for constant input and the breakthrough for a spike input is the center of mass of the peak or usually at which the peak concentration of the effluent occurs.

TABLE 1. This is an Example of all the Terms Listed in Calculations for Plotting Breakthrough Section.

Sample Number	Δ ml	cpm/ml	dpm/ml	μCi/ml	Adj. Vol. (ml)	Ce/Ci
1	23.75	18.0	20.9	5.0e-5	11.87	.005
2	24.64	266.2	309.5	6.9e-4	24.19	.069
3	25.56	770.0	895.3	2.0e-4	25.10	.02
4	26.42	1164.2	1353.7	3.0e-3	25.99	.3
5	27.33	1549.2	1801.4	4.0e-3	26.87	.4
6	28. 7	1920.4	2233.0	4.9e-3	27.70	.49 *
7	28.92	2168.2	2520.9	5.6e-3	28.49	.56

* Breakthrough point

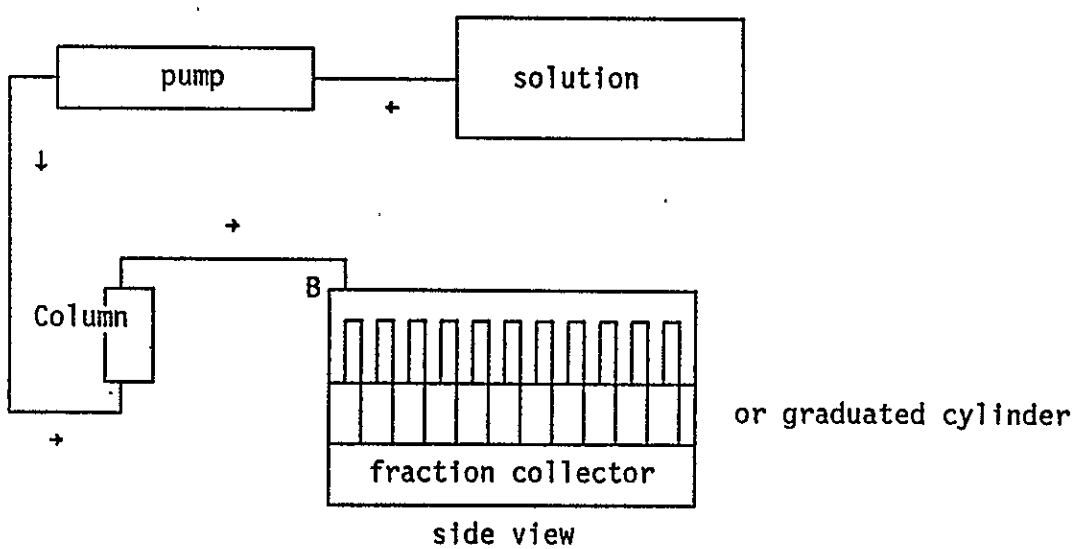

Figure 1. Diagram showing the flow of solution through the column apparatus. Effluent collection with a graduated cylinder or fraction collector.

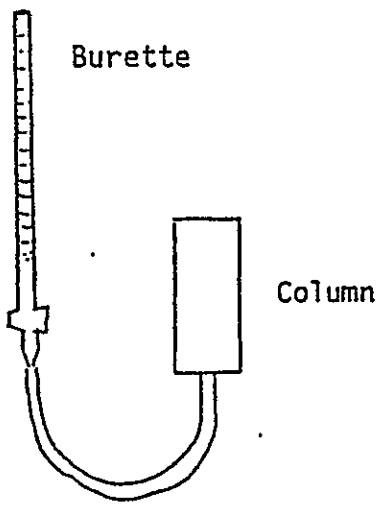

FIGURE 2. Illustration of Determining Pore Volume with a Burette

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Serne, R. J., R. C. Routson and D. A. Cochran. 1973. Experimental Methods for Obtaining Percol Model Input and Verification Data. Prepared for the U.S. Atomic Energy Commission under Contract No. AT(45-1)-1830. BNWL-1721.

Other Related Technical Procedures

G-02-SA: Surface Area Determination Ethylene Glycol Monoethyl Ether
G-03-PS: Particle Size Analysis
G-04-MC: Moisture Content Determination
G-05-PH: Measuring pH of Low-Level Radioactive Solutions
G-06-EH: Measuring Redox Potential (Eh) of Low-Level Radioactive Solutions

TEST INSTRUCTION FOR HANFORD GROUT TECHNOLOGY SATURATED COLUMN EXPERIMENT

Test Instruction No.: G-01-SC-TI- _____

Test Procedure(s) Used: _____
_____
_____

Test Material Characteristics:

Solid Material(s): _____
Influent Solution(s): _____
Other Comments: _____

_____

_____

Test Parameters:

Air temperature	_____
Approx duration	_____
Approx test start	_____
Approx test end	_____
Number of samples	_____
Number of columns	_____
Approx flow rate	_____
Approx bulk density	_____

Requested by: _____ _____
Task or subtask Leader Date

Received by: _____ _____
Staff Date

DATA SHEET FOR SATURATED COLUMN EXPERIMENT

Run no.: G-01-SC-TI-_____

Laboratory Record Book(s) Used for this test: 1._____
2._____
3._____

Solid Material Characteristics	1	2
Material type		
Weight (g)		
Particle size		
Surface Area (m^2/g)		
Moisture Content (g/g)		
Bulk density (g/cm^3)		

Influent Solution Characteristics:
Type Synthetic Untreated
Chemicals Used _____ location collected_____
_____
_____
_____
_____

Column Characteristics:
Inner diameter _____
Outer diameter _____
Length _____
Type of material _____

Balance used: Type(s) _____
ID. number(s) _____
Calibration date(s) _____

Test Parameters:
Air temperature _____
Test start _____
Test end _____
Number of samples _____

Performed by: _____ _____
Test Operator Date

Reviewed by: _____ _____
Task or subtask Leader Date

TITLE: G-05-PH: MEASURING pH OF LOW-LEVEL RADIOACTIVE SOLUTIONS

1.0 APPLICABILITY

This procedure provides step-by-step instructions for calibrating a pH electrode and for taking pH measurements of low-level radioactive solutions.

This procedure applies to pH measurements taken in support of leaching studies for the Hanford Grout Technology Program.

2.0 DEFINITIONS

Radiation Work Procedure (RWP) - This is a set of instructions for safe handling of radioactive material in the laboratory. The RWP covers a number of topics and shall be read and understood before performing any work in the laboratory.

3.0 RESPONSIBLE STAFF

- Task Leader
- Cognizant Staff

4.0 PROCEDURE

4.1 MATERIALS

pH meter
pH combination electrode 0-14 pH
Magnetic Stirrer
Stir Bars
Scintillation vials
pH buffers

4.2 SAFETY PRECAUTIONS

In using radioactive substances and/or solutions protective clothing should be used to reduce the possibility of contamination. Each laboratory is supplied with a radiation work procedure (RWP) which outlines the types and quantities of radionuclides permitted with instructions for handling. Record the number of the RWP in the laboratory record book.

QAO Approval	Approved (Line Manager)
Prepared by Wayne J Martin	Technical Review

Procedure No.	Revision No.	Date Issued	Page
G-05-PH	0	06-03-87	1 of 3

4.3 CALIBRATION

4.3.1 Review your pH meter users manual and determine if the slope of the pH meter is automatically adjusted or if it's a manual function.

4.3.2 Adjust the temperature control of pH meter to room temperature, which in most cases is approximately 25°C.

4.3.3 Check the instrument and the electrode to determine if they are properly connected and in working order. The pH electrode used is of your preference and should be both compatible with your meter and the type of solution being tested. Read the manual provided with the electrode for the necessary information. Record the serial number of the pH meter and type of electrode used in the laboratory record book.

4.3.4 In selecting the pH buffers to use for calibration, one must determine or assume the range of pH for the sample(s) to be tested. If samples are above 7 than use pH 7 and 10 and below 7 use pH 4 and 7. In some cases others buffers can be used for calibration and would be at the discretion of the user. After selecting the pH buffers record the lot numbers in the laboratroy record book.

4.3.5 Place about 10 mls of each buffer in each of two scintillation vials.

Example: 10 mls of pH 7 buffer in two scintillation vials;
10 mls of pH 4 buffer in two scintillation.

Label the vials containing the corresponding buffers as follows: test pH 4, test pH 7, rinse pH 4, and rinse pH 7.

4.3.6 Rinse pH electrode with distilled water. Blot dry with tissue. Remember that all waste must be disposed for in a radiation waste container lined with plastic.

4.3.7 Dip electrode in the rinse pH 7 buffer, then place it in the test pH 7 vial and stir. Allow the meter reading to stabilize (5 minutes). The acceptance criteria for pH measurement is a stable reading consistent to within 0.05 pH units. Once the reading has been taken, place the meter in standby.

4.3.8 Repeat steps 4.3.6 and 4.3.7 with pH 4 or 10 buffer using the temperature control to adjust the reading.

4.4 MEASURING SAMPLE

4.4.1 Sample has to be stirred during measurement with some type of stirring device such as a magnetic stirrer with a stir bar.

4.4.2 Immerse pH electrode in the sample. Allow the sample to equilibrate for about 5 minutes and record the measurement in the laboratory

record book. Use the same acceptance criteria for these readings as stated in step 4.3.7.

4.4.3 Remove the electrode from the sample and rinse electrode with distilled water from a squeeze bottle into a radioactive liquid waste container. Waste solutions will be disposed of as outlined in the RWP.

4.4.4 Blot the electrode with a tissue and throw the tissue into radioactive solid waste container.

4.4.5 Depending on the concentration of radionuclides in your solutions it maybe necessary to monitor hands, work area and sample container for any radioactive contamination between samples. Consult with the buildings Radiation Protection Technician (RPT) for appropriate instructions before performing the work.

4.4.6 Repeat steps 4.4.1 through 4.4.4 for all samples. After completing the series of samples the electrode must be rinsed with 0.1 M HNO_3 solution into a liquid waste container. Then rinse with distilled water. To store the electrode immerse in pH 4 buffer solution.

4.4.7 The work area must be cleaned thoroughly and monitored for contamination. Waste must be disposed of according to the RWP.

PNL TECHNICAL PROCEDURE

TITLE: G-06-Eh: MEASURING REDOX POTENTIAL (Eh) OF LOW-LEVEL RADIOACTIVE SOLUTIONS

1.0 APPLICABILITY

This procedure provides step-by-step instructions for calibrating a Eh electrode and for taking Eh measurements of low-level radioactive solutions.

This procedure applies to Eh measurements taken in support of leaching studies for the Hanford Grout Technology Program.

2.0 DEFINITIONS

- Radiation Work Procedure (RWP) - This is a set of instructions for safe handling of radioactive material in the laboratory. The RWP covers a number of topics and shall be read and understood before performing any work in the laboratory.

- Redox Potential (Eh) - This relates to the reduction-oxidation potential of the solution. The value is useful in understanding the speciation of selected elements for evaluating their potential solubility limits.

3.0 RESPONSIBLE STAFF

- Task Leader

- Cognizant Staff

4.0 PROCEDURE

4.1 MATERIALS

pH meter
Eh electrode
Magnetic Stirrer
Stir Bars
Scintillation vials
pH buffers
Quinhydrone $C_{12}H_{10}O_4$

4.2 SAFETY PRECAUTIONS

In using radioactive substances and/or solutions protective clothing should be used to reduce the possibility of contamination. Each laboratory is supplied with a radiation work procedure (RWP) which outlines the types and

QAO Approval: J E Thompson		Approved (Line Manager): J K Greenley	
Prepared by: Wayne J Martin		Technical Review: R Jeff Serne	
Procedure No. G-06-Eh	Revision No. 0	Date Issued 06-03-87	Page 1 of 3

quantities of radionuclides permitted with instructions for handling. Record the number of the RWP in the laboratory record book.

4.3 CALIBRATION

4.3.1 Review your pH meter users manual for proper use for direct millivolt readings.

4.3.2 Check the instrument and the electrode to determine if they are properly connected and in working order. The Eh electrode used is of your preference and should be both compatible with your meter and the type of solution being tested. Read the manual provided with the electrode for the necessary information. Record the serial number of the pH meter and type of electrode used in the laboratory record book.

4.3.3 Use the pH 7 and 4 buffers for calibration. In some cases others buffers can be used for calibration and would be at the discretion of the user. In most cases pH 4 and 7 would provide adequate values for calibration. Record the buffer lot numbers in the laboratory notebook.

4.3.4 Place about 10 mls of each buffer in each of two scintillation vials.

Example: 10 mls of pH 7 buffer in two scintillation vials;
10 mls of pH 4 buffer in two scintillation.

Label the vials containing the corresponding buffers as follows: test pH 4, test pH 7,rinse pH 4 and rinse pH 7. Place a stir bar and 2-3 grams of quinhydrone to each vial to make a saturated solution. These solutions should be prepared daily, because their shelf life is very short.

4.3.6 Rinse pH electrode with distilled water. Blot dry with tissue. Remember that all waste must be disposed of in a radiation waste container lined with plastic.

4.3.7 Dip electrode in the rinse pH 7 buffer, then place it in the test pH 7 vial and stir. Allow the meter reading to stabilize (5 minutes) and adjust the reading to its pH 7.0 millivolt equivalent. The acceptance criteria for a stable reading is ±5 mv in 5 minutes. Once the reading has been stablized place the meter in standby.

4.3.8 Repeat steps 4.3.6 and 4.3.7 with pH 4 or 10 using the temperature control to adjust the reading. Read your manual for the appropriate instructions.

4.4 MEASURING SAMPLE

4.4.1 Sample has to be stirred during measurement with some type of stirring device such as a magnetic stirrer with a stir bar.

4.4.2 Immerse Eh electrode in the sample and allow the sample to equilibrate for about 5 minutes and record the measurement in the laboratory record book. Use the same acceptance criteria with these reading as used for the buffers.

4.4.3 Remove the electrode from the sample and rinse electrode with distilled water from a squeeze bottle into a radioactive liquid waste container. Waste solutions will be disposed of as outlined in the RWP.

4.4.4 Blot the electrode with a tissue and throw the tissue into radioactive solid waste container.

4.4.5 Depending on the concentration of radionuclides in your solutions it maybe necessary to monitor hands, work area and sample container for any radioactive contamination between samples. Consult with the buildings Radiation Protection Technician (RPT) for appropriate instructions before performing the work.

4.4.6 Repeat steps 4.4.1 through 4.4.4 for all samples. After completing the series of samples the electrode must be rinsed with 0.1 M HNO_3 solution into a liquid waste container. Then rinse with distilled water. To store the electrode immerse in pH 4 buffer solution.

4.4.7 The work area must be cleaned thoroughly and monitored for contamination. Waste must be disposed of according to the RWP.

4.4.8 Record all data in labortory record book and/or on a data sheet, if provided by another procedure.

4.5 CALCULATIONS

4.5.1 In calculating the millivolt reading for quinhydrone at a specific pH use the following equation;

Eho - (pH x 59.2mv) = Eh (mv)

where

Eho = 699 mv (this is derived from pe + pH = 11.82 for quinhydrone)

TITLE: G-10-DE: PROCEDURE FOR CALIBRATING AND OPERATING A DETECTOR FOR RADIONUCLIDE DETERMINATION

1.0 APPLICABILITY

This procedure applies to the calibration and operation of detectors for radionuclide determination. The calibration shall be conducted annually or as needed on each detector. By performing this procedure the user can ensure accurate and accountable data when the detectors are producing radioanalytical investigations on unknown and known samples. It also can help in detecting error in the different programs used in the running of the calibration procedure, establishing any peak shifts in the spectra, and checking the data produced by the programs used in the manipulation of data. This procedure should be conducted by a person certified to operate the overall radiocounting system.

This procedure applies to all detectors operated by the Geochemistry Section and used for providing radioanalytical data in support of the Hanford Grout Technology Program.

2.0 DEFINITION

2.1 NBS - National Bureau of Standards - a government organization which provides standard quantities and/or units of measure that all standard sources must meet for use in analytical comparison and for deriving quantitative values.

2.2 RWP - Radiation Work Permit - this document is prepared by a certified person in radiation protection to help provide guidance in protecting the worker and the work place from possible contamination. The permit spells out the type of work, precautions, and the clothing necessary for performing radiation tasks in said facility.

3.0 RESPONSIBLE STAFF

- System Custodian
- Cognizant Staff

4.0 PROCEDURE

QAO Approval: L E Thompson	Approved (Line Manager): Robert A Kay
Prepared by: Wayne J. Martin	Technical Review: R. Jeff Serne

Procedure No.	Revision No.	Effective Date	Page
G-10-DE	0	~~April 7,~~ 4/21 1988	1 of 12

A.22

Effective Date Changed To Allow Time For Distribution After Last Signature was obtained. DH Mitchell 4/

4.1 MATERIALS

Nuclear Data Counting Facility (ND 6700)
Detector
Standard (Nuclear Bureau of Standards traceable)

4.2 SAFETY PRECAUTIONS

- Radionuclides - all samples shall be handled as outlined in the Radiation Work Permit (RWP) which shall be obtained and posted before any samples are analyzed. All unknown quantity samples must be handled as if they were radioactive. Careful consideration shall be given to the packaging of samples to avoid contamination of the detectors and/or counting chambers.

- High Voltage - all detectors require special voltages supplied by high voltage supplies and amplifiers. Care should be taken in the operation and handling of such instruments.

4.3 OPERATING PROCEDURE

- Before any operations can be performed the ND 6700 system must be in full operations with the necessary equipment checked by a qualified operator. This person will be appointed by the task leader as the system custodian. Any of the instructions outlined in this procedure are discussed in the supplied manuals for the Nuclear Data System. Throughout this section reference to the manuals are given if further detail is needed. Also the symbol <CR> will designate a carriage return and the use of the word type isn't part of the command line.

4.3.1 Sign-on; (Example; HEL 6 <CR> (password) <CR>)
- the password for the different detectors can be obtained from the system custodian.

4.3.2 Type R PARS; (operation manual 07-0108, pg. 7-37 through 54)

Set-up your sample information which will correspond to your standard. (title, identification, etc.)

4.3.3 Type LUP (Logical Unit Parameter)

Make sure DEF 8 & 12 are specific to the detector that will be in use.

4.3.4 Place the proper NBS mixed standard in the detector chamber. This standard should be in the same geometry as the unknown samples to be counted. Make sure the standard has a certified calibration and/or information sheet.

Count the standard for 10 minutes (600 sec.). This amount of time can vary with the type of standard. If the standard has a very low count rate then more time would be needed. Conversely, if the count rate is extremely high then less time would be required. In most cases, the standards are prepared in such a manner that a 10 minute count will provide a sufficient count rate for peak resolution. Make sure you have the sample on the same shelf that you will use for counting your unknown samples, because the efficiency of the counter is different at different distances from the detector.

4.3.5 Once the NBS standard is through counting you must transfer the information to disk.

Type R WRITE <CR> (ADC/GROUP): 2/1 <CR>.
(reference Vol. I, pg. 7-18 through -24)
Each detector will be numerically characterized through the ADC (Analog to Digital Converter) and your system custodian can provide the correct number for each detector in order to provide the correct response to this question.

Type R Head (reference vol. I. pg. 7-27 through -36)

Type R PEAK (reference Vol. I, pg. 7-55)

This program produces a printout containing information which can be compared to the energies listed on the NBS standard sheet under the column GAMMA RAY ENERGY. Remember to account for energy conversions if necessary (i.e. Kev and Mev).

If the peak energies of the individual isotopes are ± 1 Kev of those values specified by the certified calibration sheet, then you <u>don't</u> have to Run an energy calibration, instead skip to efficiency calibration (Step 4.3.8). If they don't meet this criterion you must run the R ENERGY next, (Step 4.3.6).

4.3.6 ENERGY CALIBRATION

Calibration of the detector can be started by using the peak search printout and manually reviewing the peaks on the screen with the NBS standards calibration sheet. Take the peak search printout which will give you the channels that correlate with the specific peak energies. Highlight the peak energies which compare to those on the calibration sheet.

4.3.7 Type R ENERGY (reference Vol. I, pg. 6-34 through -38)

This program asks for channel and energy values associated with specific peaks of interest. This information derived from the standards calibration sheet will be used. The prompt on the screen is as follows;

CHANNEL #: (enter channel number) <CR>

ENERGY #: (enter energy value) <CR>

The head program prints the sample parameters, which provides information about the sample or standard presently being analyzed. This helps in identifying each printout.

Your standard calibration sheet may include peaks outside the window setting (or region of interest) of the spectrum, therefore only include those peaks present in the energy calibration. Repeat this process until all peak energies are entered.

After you have entered all the necessary peaks hit <CR> after the CHANNEL # prompt.

It will then request the type of data fit (Linear or Quadratic) for our purposes a linear fit is adequate, enter "L".

If the file is correct and no changes are needed the file can be updated by answering Y = yes.

Once the energy calibration is completed, count the NBS standard for the same amount of time, run the R HEAD program which provides an information sheet with the sample parameters and then run the R PEAK program which provides the peak search information. Then compare the standard peaks from the calibration sheet to those from the newly generated printout. If the peaks of interest are within ± 1 Kev of those outlined on the calibration sheet the energy calibration is done and you can proceed to the efficiency calibration. If this criterion is not met rerun R ENERGY following this same procedure to fine tune the fit. If at this time the energy calibration is still not correct consult the system custodian for assistance.

4.3.8 Efficiency Calibration (Reference Vol. I, pg 6-38 through 6-41)

After successfully completing the energy calibration, collect the new peak search printout and the NBS standard calibration sheet for use later. Several geometries are used and counted in triplicate. A calibration sheet is produced for each geometry. Figure 1 and 2 are examples of energy calibration data sheets.

Here you must change your Logical Unit 11 to a specific Efficiency Table. Within the library of efficiency tables (file directory EFF) make a copy of one of the existing tables for generating a new table. The ND system recognizes efficiency tables by their particular format, therefore by making a copy and writing new

information into it the system will be able to use the your new table in the data manipulation programs. When selecting a table to copy use one which had previously been prepared for the detector you are calibrating. Consult the system custodian for this information.

Using the COPY command make the new table.

Example of the command line:

COPY EFF.TAB1, EFF.CM244 <CR>.
↑ ↑
old table new table

Now you have your efficiency table, change Logical Unit 11: Type DEF 11 EFF.CM244. Type LUP to make sure the parameters are set correctly.

4.3.9 Type R EFF

Now the system is ready for efficiency calibration. Gather together the peak search printout and the NBS standard calibration sheet, this information is needed for completing the following section.

It will ask:

DO YOU WISH TO ADD POINTS TO THE END OF THE FILE? N<CR>

At this point you don't have any data in the table and needed to make a new data set, therefore answer N = no.

DO YOU WANT AN EFFICIENCY TO BE CALCULATED? Y<CR>

DO YOU WISH TO INITIALIZE THIS FILE (Y/N) # =

Your purpose here is to create efficiencies, therefore answer Y = yes.

Enter Title for this file (64 characters max)
Enter shelf name (0-5)
Enter geometry name (4 chars max)
Enter detector name (4 chars max)
Enter certificate name (8 chars max)

Now it will ask for the standard date which is the date the standard was prepared. The standard date is on the NBS calibration standard sheet. Put this date and time in like this:

Example: 01Sep78 0900:00

Now it asks for Acquisition date of your standard sample, which would be the day you counted the standard. Enter this date and time as before (Information located on the peak search printout).

Now it asks for the set live time. Enter the time the NBS standard sample was counted for (600 sec.) Information provided on the peak search program.

Now we are ready to enter the information for each peak.

The program asks for the following information in this manner; ENERGY, HALF-LIFE, UNITS OF TIME (S,M,H,D,Y), GAMMA/SEC, AREA. These terms are defined below.

ENERGY: this is the energy peak of the specific radionuclide and information is located on the NBS calibration standard sheet under GAMMA RAY ENERGY. *Remember to convert to (Mev to Kev).

HALF-LIFE: the half-life of the radionuclide of interest and information is located on the NBS calibration standard sheet.

UNIT OF TIME: this is the unit of time the half-life is given; S = sec, M = min, H = hours, D = days, Y = years.

GAMMA/SEC: this can be calculated by multiplying the gamma/sec/g by the total weight of the standard sample located on the NBS calibration standard sheet.

AREA: the area of the peak, this information is on the peak search printout under area and next to the corresponding energy.

After you have entered all the necessary peaks, hit the <CR> after the next prompt.

Now it will ask:

DO YOU WISH TO MAKE A CORRECTION? If you haven't made any mistakes hit <CR>.

If an error was made in entering the data the program will allow you to make the necessary change by answering yes to the preceding question.

Now it asks if you want to UPDATE File? Y <CR>

If the information has been entered correctly answer Y = yes.

4.3.10 Listing & Plotting Efficiencies (reference Vol. I, pg 6-42) Type R SPLIST

Now you must specify the lower & upper energy bounds of your spectrum in units of Kev. Determine this by finding how many Kev are covered by your spectrum using the screen functions (reference to the manual for keyboard functions).

Once the bounds are set the system will need to know the increments by which to divide the spectra for plotting and listing the efficiencies. This value is arbitrary and is something left up to the operator, although if your spectrum covers a small area, about 300 Kev, then increments of 5 Kev would be sufficient, if the spectrum covers a large area (i.e. 1000-2000 Kev) then use 25 Kev as the increment.

It will ask: DO YOU WANT A PLOT? Y = yes if you want a graph of the efficiencies. This plot is helpful in viewing the slope of the efficiency curve produce from your standard.

Once the plot and/or table has been generated, review the efficiencies & graph. The efficiency curve should have a smooth slope as illustrated below. If the efficiency curve doesn't look correct consult the system custodian, it's possible the R EFF program will have to be performed again.

The efficiency table is extremely important because those values are used by other programs in calculating the concentrations of the radionuclides in your unknown samples. The equations which is used in computing radioactive concentrations and/or activities are as follows:

$$DPM = \frac{CPM}{EFF \times A.A.}$$

$$DPM \times \frac{pCi}{2.22\ DPM} = ACTIVITY\ (pCi)$$

where

DPM = disintegrations per minute

CPM = counts per minute

EFF = efficiency of the detector at that energy

A.A. = Absolute Abundance of the peak of interest

pCi = picocuries, a unit measure for activity

Other values can be derived from this information as needed, such as, concentrations uCi/liter and uCi/gram. In the following section the ND system can compute these values from the spectrum of the unknown sample and sample identification information.

4.3.11 COUNTING AN UNKNOWN SAMPLE

Prepare unknown samples for counting in exactly the same geometry as the standards used in calibration. Our 3 main geometries are 15 mL of solution in a 25 mL liquid scintillation vial, 100 mL in a 100 mL polyethylene bottle and solid samples pressed into wafer form.

Now that the detector is calibrated, you're ready for your unknown samples. Place your sample in the detector chamber at the same distance from the crystal as was used for the NBS standard sample. The counting time for the sample should be long enough to achieve a sufficient count rate for a statistical analysis of the data. The peak search program provides a 2 standard derivation analysis (i.e. 2-sigma) of the area under the peak. The analysis will establish whether or not the peak area is above the detection limit (this value is a function of the background). Once the counting time is determined enter the necessary sample identification data in the R PARS program (refer to section 4.3.2).

After the counting is finished perform the following functions.

Type R WRITE <CR> ADC/GROUP: (enter detector number) <CR>
Each detector has a number and group; consult your system custodian for the information, which will provide the correct response to this prompt.

Type R PARS this program allows you to change your particular sample information.

Type R HEAD to provides a printout of the sample identification information.

Type R PEAK this will start the peak search program for analysis of the spectra and produce a printout of the results.

Type R SPLIN (reference Vol. I, pg. 7-60)

This will revise the peak file to include your efficiency information for use in calculating quantities.

Type R NID (reference Vol. I, pg. 7-61)

This compares the peak search information to the information in the nuclide library file. The nuclide libraries are available in file NUCL and can be generated if needed. Your system custodian can assist you in preparing a new library or suggesting the appropriate existing library. After typing R NID, the data are compiled and the peaks are identified.

Type R RPTS: (reference Vol. I, pg. 7-65)

This program gives you several choices of reports, which are listed below.

```
***  NUCLIDE REPORTS  ***

INDIVIDUAL REPORTS AVAILABLE
   1.  NUCLIDE LINE ACTIVITY REPORT
   2.  UNKNOWN LINE REPORTS
        (LINES NOT LISTED IN SUMMARY)
        (LINES NOT MEETING SUMMARY CRITERIA)
   4.  NUCLIDE ACTIVITY SUMMARY REPORT
COMBINATION REPORTS AVAILABLE
   3.  REPORTS 1 AND 2
   5.  REPORTS 1 AND 4
   6.  REPORTS 2 AND 4
   7. REPORTS 1, 2 AND 4
PROGRAM EXITS IF ONLY <CR> ENTERED

ENTER REPORT TYPE(s) 1 THROUGH 7#
```

This program supplies a printout of all your data and identifies those peaks which are in the library file. The data printout can be used as a permanent record for filing.

R MDA

This program compares the Nuclide Library contents with the Nuclide Summary Report to obtain the last nuclide identification.

For each nuclide in the Nuclide Library which is not reported by the NID, the MDA calculates the minimum activity which could have been detected (given the background conditions of the spectrum being analyzed).

This calculation is based on the counts in the channels where the keylines for the isotopes would have been.

4.4 ERRORS

- The major error which might be encountered, deals with the input of data into the different files. Programs must be logged correctly in order for the computer to function (i.e. input and output). All operational errors are stated in the manuals with instructions to correct any malfunctions. If you have a question about the output, consult the systems custodian.

EFFICIENCY CALIBRATION

Geometry	File Name				Geometry	File Name			Geometry	File Name		
Count Date	Std #				Count Date	Std #			Count Date	Std #		
Counting Time					Counting Time				Counting Time			
Offset	Kev/channel				Offset	Kev/channel			Offset	Kev/channel		
Energy	Count 1	Count 2	Count 3	Ave.ct.	Count 1	Count 2	Count 3	Ave.ct	Count 1	Count 2	Count 3	Ave.ct
42.8												
86.6												
105.3												
123.1												
176.4												
248.0												
380.5												
427.9												
463.4												
591.7												
600.6												
635.9												
723.3												
873.2												
996.4												
1004.8												
1274.4												
1596.5												
Remarks					Remarks				Remarks			

FIGURE 1. Procedure G-10-DE Data Sheet

Operator_____
Date_____

EFFICIENCY CALIBRATION

Standard_____

Detector_____ Geometry_____ Date_____ Initial_____

Offset_____, Kev/channel_____, Storage name/location_____

Isotope	Kev	T 1/2	A_o value/units	A_o Date	Counting Date	T	$e^{-\lambda t}$	Abund	Dilution Factor	Corrected γ-sec	Efficiency ND	Ave. Area ND		Remarks

Reviewed by_____

FIGURE 2. Procedure G-10-DE Data Sheet

Operator_____
Date_____

TECHNICAL PROCEDURE

TITLE: ALPHA AND BETA ANALYSIS OF AQUEOUS SOLUTIONS USING A LIQUID SCINTILLATION DETECTOR

1.0 APPLICABILITY

This procedure is applicable to solution samples that contain alpha and beta-emitting and/or beta emitting radionuclides.

2.0 DEFINITIONS

2.1 Calibration Standards

2.1.1 Standard Solution - The concentration in a standard solution will be traceable to the National Bureau of Standards. Standards for the liquid scintillation counter are then made up to simulate the test solution matrix.

2.1.2 Background Standard - A background standard solution is made up to simulate the composition of the test solutions, except the background standard does not contain alpha and beta-emitting radionuclides.

2.2 Counting Efficiency (d/c) - The efficiency with which a counter detects the radioactivity (disintegrations/count) in a sample, determined with standard solutions.

2.3 cpm - Counts per minute

2.4 dpm - Disintegrations per minute

2.5 BKG - Background counts per minute determined with background standards

2.6 LRB - Laboratory record book

2.7 ABE - Alpha or Beta Emitting

3.0 RESPONSIBLE STAFF

Staff responsible for implementing this procedure are:

- Task Leader
- Scientist
- Technician

Concurrence (LINE MANAGER) 9-14-90	Date 8-27-90	Approval (TECHNICAL REVIEW) 9-14-90	Date 8/27/90
Prepared by Wayne J. Martin	Date 8-16-90	QAD Concurrence	Date 10-8-90

Procedure No.	Revision No.	Effective Date	Page
G-11-ABE	0	8/27/90	1 of 4

4.0 PROCEDURE

The concentration of ABE particles in a sample may be measured by the scintillation process. With the sample solution dissolved, dispersed, or suspended in a liquid scintillation medium, part of the kinetic energy of the alpha-emitting particles is converted into photons (light). Photons are emitted isotropically by the excited scintillation solution. As the photons move, optical reflectors in the liquid scintillation counter divert one-half the photons to each of two photomultiplier tubes. When a sufficient number of photons have been detected by the photomultiplier tube, a voltage pulse proportional to the number of detected photons will be produced as output.

4.1 Equipment and Materials

- Liquid Scintillation Counter
- Liquid Scintillation Solution or Cocktail
- 20-mL Glass Liquid Scintillation Vials
- Teflon Tape
- Calibrated Pipettes
- Liquid Scintillation Counter Manual

4.1.1 Preparation of glass scintillation vials

4.1.1.1 For higher activity samples tape the vial threads with Teflon tape. Do not allow the tape to extend below the neck of the vial.

4.2.1.2 Add 15 ± 0.5 mL of liquid scintillation to each vial.

4.2.1.3 Label the vial cap to identify the sample.

4.1.2 Preparation of the samples and standards for counting

NOTE: A triplicate set of background standards and a triplicate set of ABE standards should be prepared for each different sample matrix. Standards are prepared in exactly the same manner as the samples under investigation except 1) background standards contain no radioisotopes, and 2) ABE standards contain a known amount of ABE activity made up with a standard solution (see 2.1.1).

4.1.2.1 For all samples except the background standards, pipette an aliquot of sample (unknown solution or standard solution) into a scintillation vial prepared in 4.1.1 above, and record aliquot volume in LRB.

4.1.2.2 Tighten the appropriately labeled cap on the sample vial.

4.1.2.3 Vigorously shake each vial for 5 to 10 sec.

4.1.2.4 Upon completion of counts label print out with sample number, date, your initial and project number.

4.1.2.5 Set window settings for the isotope of interest.

4.4.2 Calculate the counting efficiency (d/c)

To calculate the counting efficiency for each type of ABE standard:

4.4.2.1 Subtract the mean background (calculated from the background standards with the same matrix as the ABE standard) from the cpm obtained from the ABE standard.

4.4.2.2 Calculate the mean of the values calculated in 4.4.2.1 for each type of ABE standard

4.4.2.3 Calculate the counting efficiency by dividing the known dpm for the ABE standards by the mean (cpm-BKG) for the ABE standards calculated in 4.4.2.2), i.e., and recorded in LRB.

$$\frac{d}{c} = \frac{dpm}{(cpm\text{-}BKG)}$$

4.4.3 Calculate the dpm/mL for the samples

4.4.3.1 Subtract the mean background (same matrix as the sample) from the cpm measured for the sample (cpm-BKG).

4.4.3.2 Calculate the dpm by multiplying the value (cpm-BKG) of the sample by the d/c of the same sample matrix.

4.4.3.3 Calculate the value dpm/mL by dividing the dpm of the sample by the sample size in milliliters.

4.4.3.4 Standard information is recorded on data sheet. Data sheets are signed, dated and with project number. These are filed or entered into log book depending upon project. Data sheets are filed under radioactive analysis. Record data in LRB.

4.5 Records

All raw counting data sheets are cross-referenced with the LRB number and page, and filed by project.

5.0 QUALITY ASSURANCE

This work will be conducted in accordance with the sponsor-approved PNL QA Manual and Program QA Plan.

6.0 REFERENCED DOCUMENTS

Beckman, LS 6800, 7800,9800 series. Liquid Scintillation System Manual.

Appendix B

100 Area Bench-Scale Soil Washing Project
Quality Assurance Project Plan (QAPjP) No. EES-084

100 AREA BENCH-SCALE SOIL WASHING PROJECT

QUALITY ASSURANCE PROJECT PLAN (QAPjP) No. EES-084

PREPARED BY

PACIFIC NORTHWEST LABORATORY
P.O. BOX 999
RICHLAND, WASHINGTON 99352

Issue Date:

Approvals:

		Dates
Project Manager	R Jeff Serne RJ Serne	03/02/93
Project Quality Engineer (Quality Assurance Officer)	ML Sours ML Sours	3/4/93
Environmental & Analytical Quality Engineering Technical Group Leader	Randy R LaBarge RR LaBarge	3/5/93
Geochemistry Section Section Manager	SA Rawson SA Rawson	3/4/93

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CONTENTS

Modifications or revisions to this QA Project Plan are discussed in Section 21, Document Control.

Distribution:

PNL

DSD Burke
HD Freeman
MA Gerber
TL Gervais
RR LaBarge (PQD File Copy)
VL LeGore
PFC Martin
SV Mattigod
RJ Serne
ML Sours

WHC

GS Corrigan
JG Field

3.0 QUALITY ASSURANCE PROGRAM

This Quality Assurance (QA) project plan applies to the Pacific Northwest Laboratory (PNL) 100 Area Bench Scale Soil Washing Activities. These activities are staffed by members of the Geochemistry Section of the Geosciences Department of the Earth and Environmental Sciences Center with support from members of Chemical Process Development Section of the Chemical Technology Department of the Materials and Chemical Sciences Center.

The QA program described here was developed to address the U.S. Environmental Protection Agency's (EPA) QAMS-005/80, Interim Guidelines for Preparing Quality Assurance Project Plans. This QA Project Plan refers to PNL's Quality Assurance Manual, PNL-MA-70.

PNL's current Quality Assurance program (PNL-MA-70) is based on ASME NQA-1-1989, Quality Assurance Program Requirements for Nuclear Facilities and meets the majority of the requirements of DOE 5700.6C. Further enhancements to the program with special emphasis on the use of Continuous Improvement (CI) processes are in progress. PNL's plan to implement the requirements of 5700.6C was submitted to DOE-RL in April 1992. The approach is to incorporate the principles of 5700.6C into PNL's Total Quality Management (TQM) initiative.

The work conducted under this Quality Assurance Project Plan has been determined to be overall PNL Impact Level II. Several Impact Level III tasks have been identified. Specific client requirements set forth in the Statement of Work #81340-92-030 will be followed.

4.0 PROJECT DESCRIPTION

The goal of this project is to conduct laboratory and bench-scale tests, perform laboratory analysis, and develop a test report to achieve the work plan milestone for the treatability tests for the 100-BC-1, and 100-DR-1 operable units.

The soils to be tested will be obtained by WHC from the 116-BC-1 and 116-D-1B trenches in the 100 area of the Hanford site. The tests to be conducted will include the physical, chemical, mineralogical characterization, wet sieving, attrition scrubbing, chemical treatment, heap leaching, and wash water treatment. The results of these tests will be used to define a complete optimized pilot scale system.

EXHIBIT 4.1. Contaminants of Concern

^{3}H	^{154}Eu
^{60}Co	^{155}Eu
^{90}Sr	^{235}U
^{134}Cs	^{238}U
^{137}Cs	$^{239/240}Pu$
^{152}Eu	Chromium

4.2 Change Control (Scope, Schedule, Budget)

Events impacting contractual elements of the project must be identified and alternatives evaluated. The Project Manager is responsible for change control, and for initiating change control actions.

Requests for changes in project scope (i.e., project objectives), schedule, or budget from that detailed in the Project Management Plan must be formally made by letter to the client. Changes to the project statement of work (i.e., project scope), schedule or budget shall required WHC concurrence via formal external change control.

Changes in QA/QC needs shall be evaluated at the time a change in scope is made.

Planned and unplanned deviations, other than changes in scope, schedule, or budget, are discussed in Section 15.2 of the QA Project Plan.

5.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

Line authority, Quality Assurance authority and support within PNL, and interfaces with Westinghouse Hanford Company (WHC) are shown in Exhibit 5.1.

Changes to organizational/interface structures shown in Exhibit 5.1, with exception of the Project Manager, that do not reflect a change in the overall scope of the activities or a change of requirements will not require a QA project plan revision but will be incorporated in the next required revision of the QA Project Plan.

The responsibilities of key PNL personnel are summarized in Table 5.1.

EXHIBIT 5.1 Organizational Interfaces

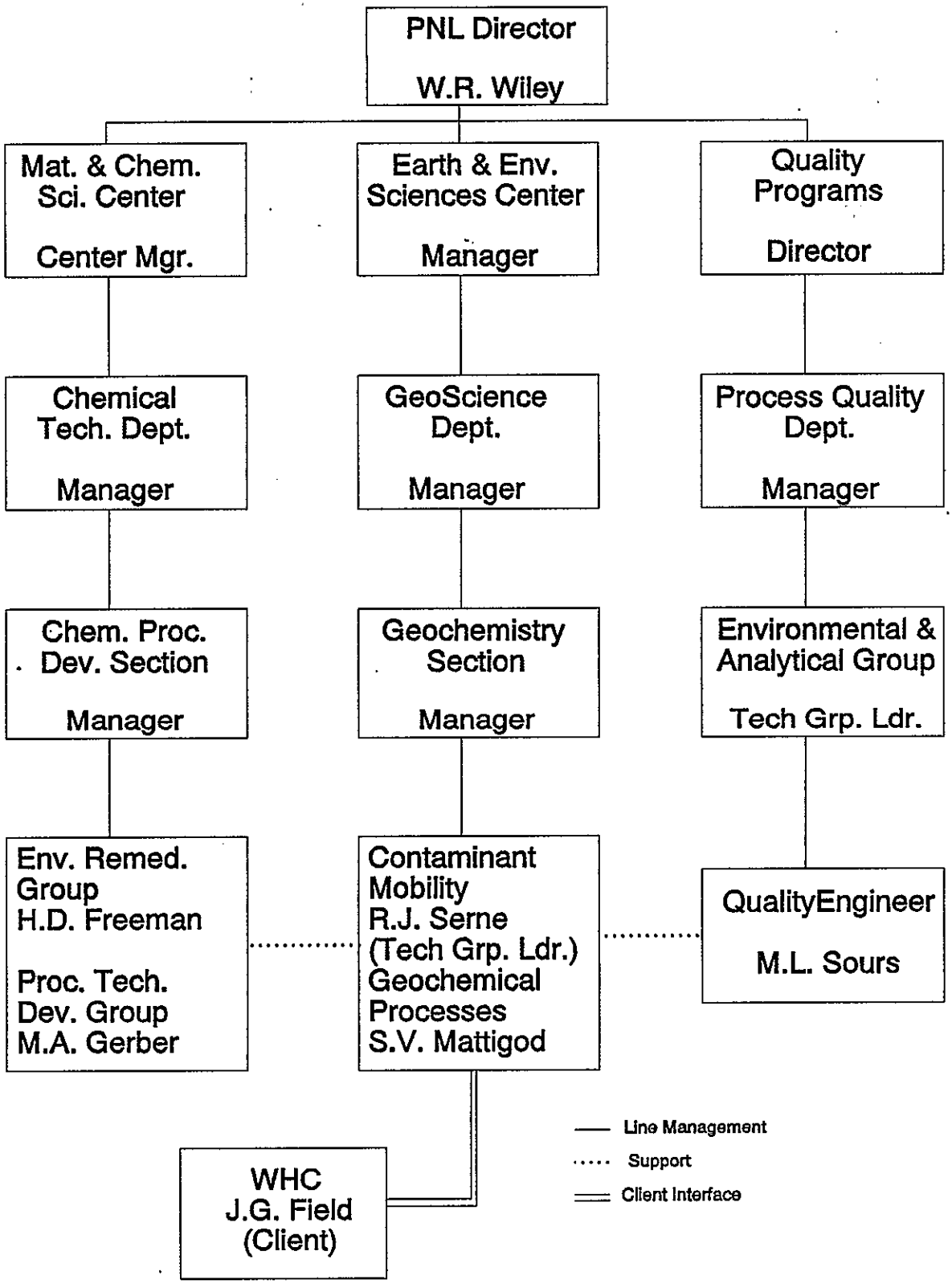


TABLE 5.1. Responsibilities of Key Personnel

Personnel	Responsibilities
Geochemistry Section Manager	Provides management review of the project. Section assures appropriate and qualified staff are available.
Project Manager (RJ Serne)	Interfaces with WHC project lead and provides weekly reports of activities. Provides overall PNL direction of the project and day-to-day activities necessary to accomplish all project objectives. Ensures that the QA project plan is prepared and implemented to and that data, QA information, and reports are produced in a timely manner. Has direct contact with the PNL Quality Engineer. Coordinates all Quality Control (QC) activities including the scheduling, preparation, and submittal of QC samples to PNL laboratories, and evaluates the results. Interacts with the Sample Analysis Task Leader to investigate suspect results.
Quality Engineer (Quality Assurance Officer) (ML Sours)	Transmits documents and records to WHC at project completion. Provides the Project Manager with QA requirements interpretation and implementation assistance. Provides for Quality Assurance training as necessary. Provides for independent quality assurance reviews, surveillances, and data quality and traceability audits. Is responsible for reviewing and has sign-off authority for QA project plans. Provides QA reports to Project Manager.
Quality Engineering	Provides independent Quality Assurance Group Leader reviews, surveillances and data quality traceability audits. Is responsible for reviewing and has sign-off authority for QA Project Plans.
Senior Research Scientist Scientist (Geochemical Processes)	Prepares QA project plan in coordination with PNL Quality Engineer. Prepares parts of the Experimental Test Plan. Interacts with lab personnel and directs the lab operations. Analyzes corrected data in collaboration with the Project Manager.
Senior Research Scientist (Chemical Process Development Section)	Prepares portions of the Experimental Test Plan and interacts with lab specialists to assure lab testing is performed correctly. Aids in interpretation of data generated and technical report preparation in collaboration with Project Manager.

6.0 DATA QUALITY OBJECTIVES

Data uses and needs along with performance goals and decisions to be made with the data generated by this project can be found in the 100 Area Soil Washing Treatability Test Plan, Draft A, Sections 2.1 and 2.2. This section of the QAPjP addresses the performance criteria: precision, accuracy, completeness, comparability and representativeness (PARCC).

Detection Limits and performance levels to be attained for the analytes of interest can be found in the 100 Area Soil Washing Bench-Scale Test Procedures.

6.1 DQO Definitions

ACCURACY - a measure of the bias of a system or measurement. It is the closeness of agreement between an observed value and an accepted value.

For this project, accuracy of chemical analyses will be determined through the analysis of matrix spikes, tracers, carriers and standard reference material (SRM), as appropriate for the sample being analyzed and as dictated by the technical procedure being used. SRMs are materials that have been certified by a recognized authority (e.g., National Institute of Standards and Technology) and which are treated and analyzed as an actual sample. When appropriate, matrix spikes will be performed by adding a known quantity of target analytes into a sample and preparing and analyzing the sample the same as a regular sample.

For measurements where matrix spikes and/or SRMs are used, percent recovery shall be used.

$$\%R = 100 \times \frac{S-U}{C_{sa}}$$

%R=percent recovery
S=measured concentration in spiked aliquot
U=measured concentration in unspiked aliquot
C_{sa}=actual concentration of spike added

PRECISION - a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions.

For this project, measures of analytical precision will be determined by the analysis of laboratory duplicates. Laboratory duplicates will be prepared by homogenizing and splitting a sample in the laboratory, and carrying the subsamples through the entire analytical process. Precision can be expressed in terms of the relative percent difference (RPD).

$$RPD=\frac{(C_1-C_2)}{[(C_1+C_2)/2]}\times 100$$

RPD=relative percent difference
C_1=larger of the two observed values
C_2=smaller of the two observed values

COMPLETENESS - a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

$$\%C=100x\frac{V}{n}$$

V=Number of Valid Data Points Acquired
n=Total Number of Data Points

Refer to Table 6.1 and 6.2 for completeness objectives.

DETECTION LIMIT - Detection limit is the minimum concentration of a substance that can be measured and reported. Method Detection Limit (MDL) is the minimum concentration of a substance that can be identified, measured, and reported with 99 percent confidence that the analyte concentration is greater than zero.

The analytical lab will be required to demonstrate the ability to meet a Practical Quantitation Limit (PQL) using recognized procedures for detection limit determination (i.e. 40 CFR 136, Appendix B or USEPA CLP SOW 3/90 or current).

Detection limits required shall be passed on to the analytical laboratory via the Statement of Work. These detection limits shall be five times lower than the performance levels stated in the 100 Area Soil Washing Treatability Test Plan, section 1.3.1.2. By specifying a detection limit of five times below the performance level, the detection limit will be easily attainable on a routine basis and will not involve additional cost.

For radiation chemistry the Minimum Detectable Activity (MDA) is appropriate. The MDA is the minimum activity detected for a specific analysis
given the specific sample size and counting time. The sample size and counting time to achieve the required MDA for this project shall be passed on to the analytical lab via the Statement of Work. The MDA will be five times lower than the performance levels stated in the 100 Area Soil Washing Treatability Test Plan, section 1.3.1.2.

MDL is defined as follows:

$$MDL = t_{(n-1,\ 1-\infty=0.99)} = xS$$

MDL = method detection limit
S = standard deviation of the replicate analyses
$t_{(n-1,\ 1-\infty=0.99)}$ = Students' t-value appropriate to a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom

REPRESENTATIVENESS - Expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness will be addressed primarily in the sample design, through the selection of sampling sites and procedures. WHC will conduct this portion of the project. Representativeness also will be ensured by the proper handling and storage of samples. Representativeness of samples selected for analysis shall be addressed in section 2.1 of the 100 Area Soil Washing Bench-Scale Test Procedures. Representativeness of data will be discussed, when appropriate, in deliverable reports.

COMPARABILITY - expresses the confidence with which one data set can be compared to another.

Comparability for this project will not be quantified, but will be addressed through the use of accepted laboratory methods. The use of standard reporting units also will facilitate comparability with other data sets. Comparability between spike recoveries between batches will be analyzed for possible recovery corrections. Comparability of other data will be discussed, when appropriate, in the final report.

6.2 Corrective Action for Results Outside Established DQOs

Results outside the established criteria in Tables 6.1, and 6.2 shall be brought to the attention of the Task Leader, Project manager and the Program Manager who shall determine and document the appropriate corrective action. These actions may include, but are not limited to, review of data and calculations, flagging of suspect data or re-analyses of individual or entire batches of samples.

6.3 WHC/Regulator Agreement Regarding Data Quality Objectives

Specific agreements regarding the development of Data Quality Objectives (DQOs) and the use of specific procedures to attain those DQOs have been made between the Regulators and WHC Program Management. Further information regarding the development of the DQOs and the specific procedures to be employed can be found in Section 4.3.4 and Appendix A, p. A-5, Section 3.2, paragraph 2 of the WHC 100 Area Soil Washing Treatability Studies Test Plan, Draft A.

Table 6.1: Data Quality Objectives for Chemical Analyses					
Characterization of Trench 116-C-1 and 116-D-1B Sediments					
Analytes	EPA Analytical Level	Analytical Method*	Data Quality Objectives		
			Relative Percent Difference (RPD)	Percent Recovery (%)	Complete
^{3}H	III	PNL-ALO-441, 443	≤ 35%	50-150%	90%
^{60}Co	III	PNL-ALO-105, 464	≤ 35%	75-125%	90%
^{90}Sr	III	PNL-ALO-106, 465, 463	≤ 35%	75-125%	90%
^{134}Cs	III	PNL-ALO-105,464	≤ 35%	75-125%	90%
^{137}Cs	III	PNL-ALO-105,464	≤ 35%	75-125%	90%
^{152}Eu	III	PNL-ALO-105,464	≤ 35%	75-125%	90%
^{154}Eu	III	PNL-ALO-105,464	≤ 35%	75-125%	90%
^{155}Eu	III	PNL-ALO-105,464	≤ 35%	75-125%	90%
^{235}U	III	PNL-ALO-101, 280, 282	≤ 35%	75-125%	90%
^{238}U	III	PNL-ALO-106, 466, 468	≤ 35%	75-125%	90%
$^{239/240}Pu$	III	PNL-ALO-106, 466, 468	≤ 35%	75-125%	90%
Cr	III	PNL-4O.48, Rev. 1	≤ 20%	75-125%	90%

*The complete title of these analytical methods can be found in Table 6.3 of this QAPjP

Table 6.2 Data Quality Objectives for Physical Tests

Step		EPA Anal. Level	Method	Data Quality Objectives
1	Sediment Preparation	III	1. ASTM D 421-85 Standard Practice for Dry Preparation of soil samples for Particle size analysis and determination of soil constants. 2. Perform analyses listed in Table 6.1 as appropriate.	Recovery of dry, well homogenized soil sample to within 5% of total unmixed as received soil sample weight after correcting for moisture content.
2	Wet Screen/Reanalysis vs Particle Size	II	1. ASTM D422-63 Standard Method for Particle-Size Analysis of Soils. Modified for wet sieving. 2. PNL Test Procedure 7-40.48 Rev. 1. 3. ^{90}Sr, perform analyses listed in Table 6.1 as appropriate	Recovery of soil sample fractional weights to within 25% of total soil sample weight. Sum of contaminant recovered from soil and extractant will be within 25% of the measure found in soil sample prior to wet sieving (values from Step 1).
3	Soil Petrography/Selective Extraction	II	1. Methods of Soil Analysis, Part 1. Chap. 8. Petrographic Microscope Techniques. 2. Electron Microscopy of Soils and Sediments: Techniques. Smart, P., and Tovey, N.K., Clarendon Press, Oxford. 1982	Sum of contaminant recovered from soil and extractant will be within 25% of the measure found in soil sample prior to chemical extraction (applies to methods 5, 9 and 10). There are no applicable standards or data quality objectives for other characterization techniques.

Table 6.2 Data Quality Objectives for Physical Tests

Step		EPA Anal. Level	Method	Data Quality Objectives
3 Cont			3. Methods of Soil Analysis Part 1. Chap 21. Water Content. 4. ASTM D854-83 Standard Test Method for Specific Gravity of soils. 5. Sequential Extraction Method, Environmental Science & Technology 23, 1989 1015-1020. 6. Methods of Soil Analysis Part 1. Chap. 12. X-ray diffraction of techniques. 7. Density Separation. Fuel Vol. 62 1983, 927-931. 8. Method of Soil Analysis Part 2. Chap. 9 Exchangeable Cations. 9. PNL Technical Procedure PNL-ALO-280, Inductively Coupled Plasma-Mass spectrometric (ICP-MS) Analysis. 10. Perform analyses listed in Table 6.1 as appropriate. 11. TOC (ASTM-D-4129-88 See references section) 12. TCLP EPA Method 1311	

Table 6.2 Data Quality Objectives for Physical Tests

Step		EPA Anal. Level	Method	Data Quality Objectives
4	Attrition Scrubbing	II	1. Attrition Scrubbing Procedure (see Bench-Scale Test Procedures). 2. Will use G-10-DE Procedure for calibrating and operating detector for Radionuclide Detection. 3. Perform analyses listed in Table 6.1 as appropriate.	Recovery of soil sample fractional weights to within 25% of total soil sample weight. Sum of contaminant recovered from soil and wash water will be within 25% of measure found prior to attrition.
5	Chemical Extraction	II	Chemical Extraction Procedure (see Bench-Scale Test Procedures): 1. Will use G-10-DE Procedure for calibrating and operating detector for Radionuclide Detection 2. Chemical Extraction Procedure based on G-06-EH, Measuring Redox Potential of low level radioactive solutions and G-05-PH, measuring pH of low level radioactive solutions. 3. Perform analyses listed in Table 6.1 as appropriate.	Sum of contaminant recovered from soil and extractant will be within 25% of the measure found in soil sample prior to chemical extraction.

Table 6.2 Data Quality Objectives for Physical Tests

Step		EPA Anal. Level	Method	Data Quality Objectives
6	Optimize Combined Extraction / Attrition/Separation	II	Attrition Scrubbing Procedure: 1. Will use G-10-DE Procedure for calibrating and operating detector for Radionuclide Detection 2. Chemical Extraction Procedure based on G-06-EH, Measuring Redox Potential of low level radioactive solutions and G-05-PH, measuring pH of low level radioactive solutions. 3. Perform analyses listed in Table 6.1 as appropriate.	Recovery of soil sample fractional weights to within 25% of total soil sample weight. Sum of contaminant recovered from soil and extractant will be within 25% of the measure found in soil sample prior to chemical extraction.
7	Heap Leach	II	Heap Leach Procedure (see Bench-Scale Test Procedures): 1. Will use G-10-DE Procedure for calibrating and operating detector for Radionuclide Detection 2. Heap Leach procedure based on G-01-SC, Procedure for Conducting Saturated Column Experiments 3. Perform analyses listed in Table 6.1 as appropriate.	Sum of contaminant recovered from soil and extractant will be within 25% of the measure found in soil sample prior to chemical extraction.
8	Water Treatment	II	To be decided. The optimal water treatment procedure will be based upon data received from steps 4-7 of Table 6.2. This planned deviation will be documented according to section 15.2, Deviations from Procedures of Requirements. Will measure appropriate contaminants, see Table 6.1.	Sum of contaminant removed from water and residual left in water will be within 25% of the measure found in water prior to treatment.

Table 6.3 Analytical Method Titles

PNL-ALO-101(Rev.1)	Acid Digestion for Metals Analysis
PNL-ALO-105(Rev.0)	Procedure for Preparation of Samples to be Counted by Gamma-Ray Spectroscopy
PNL-ALO-106(Rev.0)	Acid Digestion for Preparation of Samples for Radiochemical Analysis
PNL-ALO-280(Rev.0)	Inductively Coupled Plasma-Mass Spectrometric (ICP-MS) Analysis
PNL-ALO-282(Rev.0)	Determination of Uranium Concentration/Isotopic Composition Using ICP-MS
PNL-ALO-441(Rev.0)	Radionuclide Separation and Analyses Procedure for Tritium
PNL-ALO-443(Rev.0)	Liquid Scintillation Counting Procedure for Tritium
PNL-ALO-463(Rev.0)	Beta Counting Procedure
PNL-ALO-464(Rev.0)	Procedure for Gamma Counting and Data Reduction in the Low-Level Counting Room, 329 Building
PNL-ALO-465(Rev.0)	Strontium-90 Analysis (Oxalate-Nitric Acid Method)
PNL-ALO-466(Rev.0)	Procedure for Plutonium Separation and Initial Americium Separation by Anion Exchange
PNL-ALO-468(Rev.0) onto Counting Disks	Procedure for Electroplating Plutonium, Americium and Uranium
PNL 7-40-48(Rev.1)	Procedures and Quality Control for Energy Dispersive X-Ray Fluorescence Spectroscopy Using the BFP Approach with the KEVEX 0810A System

7.0 SAMPLING PROCEDURES

7.1 Sample Site Selection and Collection

Westinghouse Hanford Company (WHC) will be responsible for site selection and the collection of samples. As stated in the 100 Area Soil Washing and Treatability Test Plan (DOE/RL-92-51 Draft A) section 4.0.

Soil from trenches 116-D-1B and 116-C-1 (total of 50 gallons from each trench packed in 5 gallon containers respectively) will be delivered to PNL for treatability testing by WHC.

7.2 Laboratory Sample Selection

The sample homogenization and selection for characterization and further testing (steps 2-7 in Table 6.2) will be done according to ASTM D 421-85 as referenced in Step 1 of Table 6.2.

8.0 SAMPLE CUSTODY

8.1 Soil Sample Chain-of-Custody

The chain-of-custody for soil samples submitted for radionuclide and hazardous constituent analysis, as well as for Bench Scale Testing will be initiated by WHC. Additional chain-of-custody measures following receipt of the sediments at PNL, shall be controlled in accordance with **PNL-MA-567, Procedure AD-4, Sediment Sample Chain-of-Custody** and **PAP-70-801, Identification and Control of Test Materials (Testing and Analysis).**

8.2 Corrections to Documentation

If an error is made on any field or laboratory documentation, an individual may correct the error by drawing a line through the error and entering the correct information. The error shall not be obliterated. All non-editorial corrections shall be initialled and dated.

9.0 CALIBRATION PROCEDURES AND FREQUENCY

All measurement and test equipment (M&TE), for which PNL is responsible, must be controlled in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1201, Calibration Control System.**

Category 1 M&TE is calibrated by an approved metrology organization. All organizations providing Category 1 calibration services must be evaluated by the PNL Process Quality Department in accordance with PNL-MA-70 Quality Assurance Procedure **QAP-70-701, Preaward Evaluations/Surveys,** before being utilized.

Category 2 M&TE is calibrated by the user. Requirements for documenting user calibration of Category 2 M&TE are included in PNL-MA-70 Administrative Procedure **PAP-70-1201, Calibration Control System.**

Any analytical lab performing work will be designated in the Statement of Work as responsible for calibration of analytical equipment. Category 3 M&TE is not calibrated but is performance checked in the field and is for indication only. Performance checks are recorded on the Field Record Form.

10.0 ANALYTICAL PROCEDURES

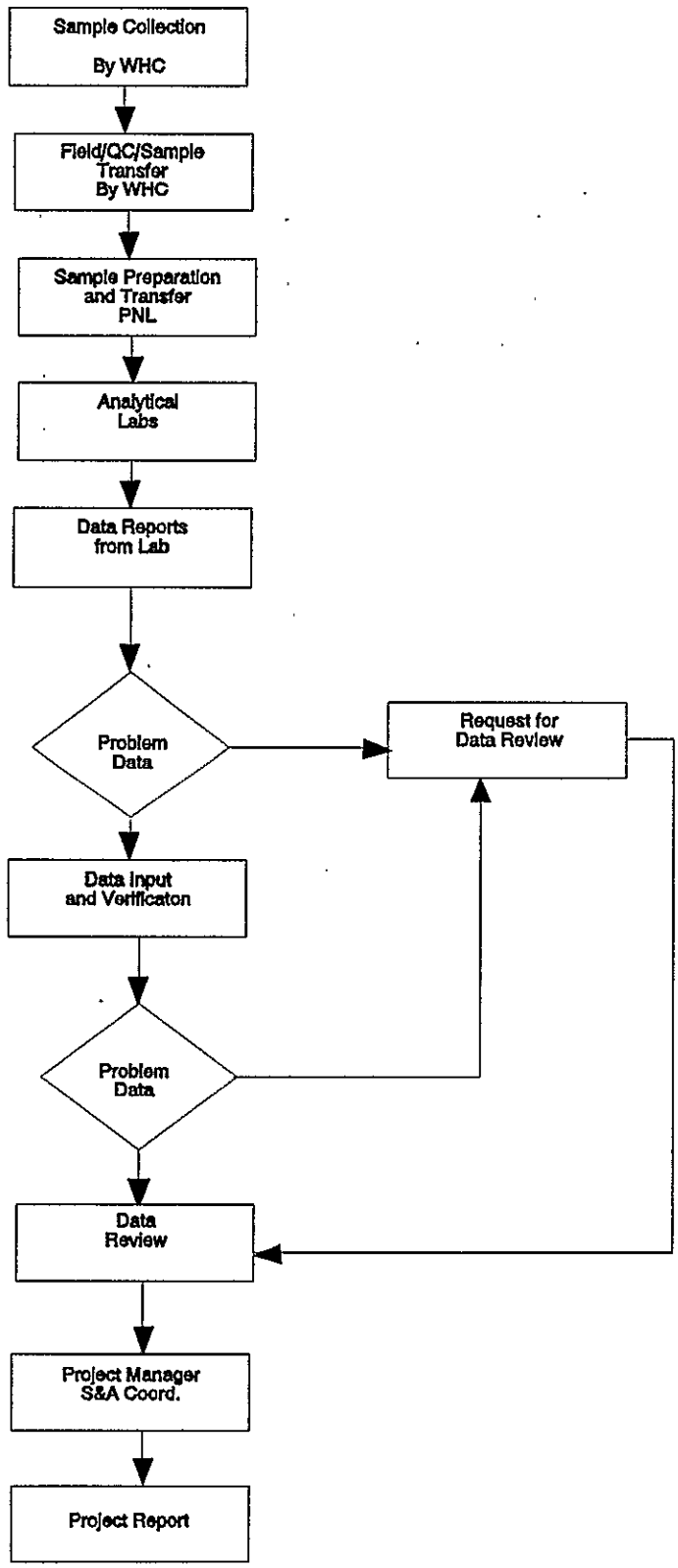
Characterization Analyses: Initial chemical constituents to be analyzed for, as well as the corresponding standard analytical methods on which the primary analytical laboratory bases its procedures are shown in Table 6.1.

Laboratory Bench Scale Tests: The bench scale physical and chemical methods are shown in Table 6.2. Upon completion of tests described in Table 6.2, the soils and extractants will be analyzed for chemical constituents using methods in Table 6.1 and Table 6.2.

11.0 DATA REDUCTION, VALIDATION, AND REPORTING

Exhibit 11.1 presents the data reduction, validation, review, and reporting process in flow-chart format. The following sections briefly describe the data reduction, validation, and reporting Procedures that shall be used for the characterization of Trenches 116-C-1 and 116-D-1B sediments. Some specific data validation methods are described in Section 12 as part of the required internal QC.

EXHIBIT 11.1



11.1 Data Management Procedures

All completed data packages shall be reviewed and approved by the Project Manager before submittal to WHC.

Analytical results for the characterization samples analyzed in Task 1 must be managed in accordance with PNL-MA-567 Procedure **DM-1, Analytical Data Handling and Verification Procedure.**

11.2 Process for Handling Suspect or Unacceptable Data

When the initial data review identifies suspect data, that data must be investigated to establish whether it reflects true conditions or an error. The investigation must be documented using a Request for Data Review (Exhibit 11.2). The Sample Analysis and Coordination Task Leader shall issue RDR numbers and maintain a log of all RDRs generated identifying their status (i.e., date issued, and date closed).

If a data value is determined to be in error, the source of the error must be investigated, the correct value established if possible, and the erroneous value replaced with the correct value. If the investigation concludes that the data are suspect (possibly in error) but a correct value cannot be determined, the data must be flagged in the comments column to indicate its suspect status.

If the source of the error was noncompliance with an established requirement or procedure, a Deficiency Report (DR) must be generated in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1502, Controlling Deviations from QA Requirements and Established Procedures.** If the source of error was due to the nonconformance of an item, then a Nonconformance Report (NCR) must be generated in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1501, Nonconformance Reports.** As a minimum, the Project Manager, Sample Analysis and Coordination Task Leader, and the Quality Engineer must be copied on the data investigation documentation (RDR). Nonconformance reports shall be sent to the WHC cognizant engineer and cognizant QAE for disposition concurrence prior to initiating the disposition.

EXHIBIT 11.2

1 ***Request for Data Review (RDR)*** ***No.:*** _____

Originator: _____ Date: _____ Phone #: _____ MSIN: _____

Project: _____ Manager: _____ Phone #: _____ MSIN: _____

Sample #: _____ Well #: _____ Collection Date: _____

Constituent: _____ Value: _____ Other: _____

2 ***Reason for Review***

3 ***Data Review Findings***

Reviewer: _____ Date: _____ Attachments: _____

4 ***Response/Action***

Laboratory Coordination: _____ Date: _____

Signature When Complete

5 ***Data Base Management Action***

Data Base: _____ Date: _____

Signature When Complete

6 ***RDR Closure***

_____ Date: _____

Laboratory Coordination Task Leader Signature

_____ _____

Originator's Signature Date Quality Engineer Signature Date

7 ***Distribution***

Originator Project Manager Data Management Reviewer
Laboratory Coordination RDR Logbook (original) Quality Engineer

ADDITIONAL DISTRIBUTION AFTER CLOSURE:

12.0 INTERNAL QUALITY CONTROL CHECKS

12.1 Physical and Chemical Test Quality Control Checks

Laboratory Bench Scale Tests: Quality Control (QC) checks for the Bench Scale tests that require analysis of the contaminants of concern include the analysis of blind duplicates every 10 samples and the analysis of blank samples when applicable. In addition, QC checks for the chemical analyses indicated in Tables 6.1 and 6.2, respectively, are specified in the test method or procedure.

Characterization Analyses: The requirements for an internal laboratory QC program that is implemented through the laboratory's analytical procedures will be passed to the Analytical Chemistry Laboratory (ACL) via Statement of Work (SOW).

QC checks for the chemical analyses indicated in Table 6.1 to be performed in the ACL are specified in the test method or procedure.

12.2 Acceptable Limits/Results Requiring Action

The acceptance limit for blind standards is ± 2 standard deviations (s.d.). In inter-laboratory comparisons using actual field samples, difference between laboratory results of 2.8 s.d is allowed. This criterion is based on the reproducibility limit, with 95% confidence that random error is not responsible for the difference.

13.0 PERFORMANCE AND SYSTEM AUDITS

Surveillances are, in a sense, mini-audits that provide the project manager with the ability to view the status of the project on a more frequent, snapshot-in-time, basis. In addition, they provide a cost effective means to view a wide range of project and analytical processes.

Compliance, real-time, and data traceability surveillances are performed by Quality Engineers of the Quality Verifications Department (QV). Compliance surveillances are performed to ensure that a specific requirement, or set of requirements, is being implemented. Real-time surveillances are performed during the work or analytical process to ensure that specific standardized procedures are being implemented. Data traceability surveillances are performed to ensure that the resultant project data are traceable back through the analytical process, through sample handling and transportation, back to the date, time, location, staff, and technique used to collect the sample. Surveillances are performed in accordance with PNL-MA-70 Quality Assurance Procedure **QAP-70-1001, Planning and Performing Surveillance.**

System audits, or simply audits, are performed by the PNL Quality Verification Department on a periodic basis. Audits are planned and performed in accordance with PNL-MA-70 Quality Assurance Procedure **QAP-70-1801, Internal Audits.** Quality Assurance audit personnel are qualified in accordance with PNL-MA-70 Quality Assurance Procedure **PAP-70-204, QA Audit Personnel Qualification.**

A minimum of three (3) surveillances will be performed during the life of this project. The results of surveillances and audits will be made available to project and line management as well as to individuals contacted.

PNL does not currently have a Laboratory-wide system in place for Performance Audits. This matter is currently being addressed.

For the Characterization Task (Task 1), PNL participates in, and will rely on the following performance evaluation (PE) programs:

1) USEPA Water Pollution (WP) Assessment for ICP Analysis--semi-annual performance evaluation samples are analyzed and reported,

2) CLP Performance Evaluation Program for Inorganics--quarterly performance evaluation samples are analyzed and reported,

3) USEPA Nuclear Radiation Assessment Program for radioactive isotopes--Evaluation via this program occurs on a continuous basis throughout the year with an average of two or three different performance evaluation samples received, analyzed and reported each month.

The results of these Performance Evaluations shall be requested from the participating labs via the Statement of Work and will be included in the Quality Assurance reports to the Project manager (see Section 16).

14.0 PREVENTIVE MAINTENANCE

Routine equipment and facility maintenance and instrument services ensure the timely and effective completion of a measurement effort. Analytical laboratory equipment maintenance is the responsibility of the manager of the analytical laboratory.

Analytical laboratories shall have sufficient critical spare parts on hand or backup instrumentation available to prevent any delays in work.

A list of critical analytical instrumentation and spare parts and the backup instrumentation available shall be addressed in a memo to the project files. Requirements for a Critical Spare Parts & Instrumentation List will be passed to the participating laboratory via the Statement of Work (SOW).

15.0 CORRECTIVE ACTION

Corrective action must be initiated by the Project Manager or cognizant Task Leader when unplanned deviations from procedural, contractual or regulatory requirements occur. The need for corrective action may be revealed by observations of measurement system response, during data reasonableness checks (brief comparison of newly collected data against observed historical trends), when discrepancies are noted during instrument calibration, or during data analysis.

15.1 Measuring and Test Equipment (M&TE) Calibration Discrepancies

Instruments or equipment found to be operating outside acceptable operating ranges (as specified in the applicable technical procedure or manufacturer's instructions) must be investigated. A Calibration Discrepancy must be initiated in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1201, Calibration Control System**, when it is determined that M&TE is not within calibration and that data have been collected after the calibration expired.

15.2 Deviations from Procedures or Requirements

Unplanned deviations from procedural, contractual, or regulatory requirements must be documented by completing a Deficiency Report (DR) in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1502, Controlling Deviations from QA Requirements and Established Procedures.** The DR must identify the requirement deviated from, the cause of the deviation, whether any results were effected, and corrective action needed to remedy the immediate problem and to prevent recurrence.

Planned deviations, documented (including justification) and approved by the Project Manager or Task Leader in advance, do not constitute a deficiency as defined in PAP-70-1502 and do not require development of a DR.

15.3 Corrective Action for Significant Conditions Adverse to Quality

When significant conditions adverse to quality are identified, the cause of the conditions and the corrective action taken to preclude repetition will be documented and reported to immediate management for review and assessment by a Corrective Action Request (CAR) in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1602, Corrective Action.** "Significant" conditions are identified in Section 4.2.1.1 of PAP-70-1602.

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The project Quality Engineer shall prepare a monthly report to the Project Manager. This report shall include, as a minimum, the results of all surveillances and audits with recommended solutions to any problems. The results of performance evaluation samples shall be reported to the Project Manager as they are received.

QA/QC reports to the Laboratory Manager shall include an assessment of data accuracy, precision and completeness. Copies of these reports shall be forwarded to the Project Manager.

Deviations from this QA project plan, as well as the results of surveillances and audits, must be documented, described and reported to the Project Manager. Quality Assurance related information must be reviewed by the cognizant PNL Quality Engineer.

Problems identified by project personnel must be reported to the project manager immediately for resolution. Problems involving data quality or sample integrity, must be thoroughly documented.

Line management must be included on the distribution of all audit reports. Significant problems encountered in day-to-day operations must be reported to line management immediately by the Project Manager.

17.0 RECORDS

17.1 Records Management

Project records must be indexed and maintained in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1701, Records System.** A Records Inventory and Disposition Schedule (RIDS) must be prepared and submitted for review and approval by the Records Specialist and Quality Engineer. Records retention schedules shall be based on DOE Order 1324.2A, Records Disposition, and applicable regulatory requirements.

- The Project Manager must assure that documents are reviewed for technical adequacy, accuracy, and completeness to verify that the documents support fitness for operation and conformance to specifications and procedures.

- Any problems or deficiencies noted in the records must be properly resolved and documented in accordance with PNL's deficiency/nonconformance system (see Section 20).

17.2 Turnover of Records To WHC

If this project is not completed within 12 months, project records will be transferred to the PNWD Records Center at least annually. Within 30 days after project completion, all remaining records will be transferred to the PNWD Records Center. All PNL generated record copy, Quality affecting documents shall be transmitted to WHC within 90 days after completion of the project. These activities must be coordinated through the PNL Records Specialist.

18.0 PROCUREMENT CONTROL

18.1 Purchase Requisitions and Subcontracts

Procurement of items and subcontracted services are governed by PNL-MA-70 Administrative Procedure **PAP-70-401, Preparation, Review, and Approval of Purchase Requisitions.**

18.2 Work Orders and Work Package Authorizations

Work Package Authorizations (WPAs) or Work Orders (WOs) to individuals or groups outside the project organization must be generated and issued in accordance with PNL-MA-70 Administrative Procedure **PAP-70-404, Obtaining Services Via Work Orders.** As appropriate (as specified in PAP-70-404), a letter of instruction (LOI) or statement of work (SOW) must accompany each WO or WPA.

19.0 STAFF TRAINING

Staff performing activities affecting quality shall be issued documented training assignments including applicable administrative and technical Procedures and this QA project plan, according to **PAP-70-201, Indoctrination and Training.** Documentation of training shall be maintained by Laboratory Training.

Requirements for the training of analytical staff to the procedures or methods to be performed shall be passed to the analytical laboratories via Statement of Work in accordance with Section 18.0, Work Orders and Work Package Authorizations.

20.0 NONCONFORMANCES AND DEFICIENCIES

For materials found to be in nonconformance with specifications, a Nonconformance Report (NCR) must be generated and the item(s) dispositioned in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1501, Nonconformance Reports.**

Unplanned deviations from Procedures, plans, specifications, or related documents shall be documented using a Deficiency Report (DR) in accordance with the requirements in PNL-MA-70 Administrative Procedure **PAP-70-1502, Controlling Deviations from QA Requirements and Established Procedures.** Potentially impacted data shall be segregated or flagged by the project manager pending evaluation of the deficiency's impact on the data and final disposition of the DR.

See also Section 11, Data Reduction, Validation and Reporting, for handling suspect or unacceptable data and Section 15, Corrective Actions, for corrective actions.

21.0 DOCUMENT CONTROL

21.1 QA Project Plan Control

Distribution and control of this QA project plan shall be performed in accordance with PNL-MA-70 Administrative Procedure **PAP-70-205, Quality Assurance Plans.**

Modifications to this QA project plan shall be made in accordance with Section 4.6 of PNL-MA-70 Administrative Procedure **PAP-70-205, Quality Assurance Plans**, that is, either by revision or by issue of an Interim Change Notice (ICN). Any PNL staff member may request an interim change to this QA project plan at any time by submitting a Document Change Request (DCR) to the Project Manager or Quality Engineer. Changes in scope, schedule or budget are addressed in Section 4.2 of this QAP.

21.2 Technical Procedure Control

Many of the technical procedures referenced by this QA project plan are contained in PNL-MA-567, Procedures for Ground-Water Investigations, WAC 173-303, WAC 173-340, and WHC-CM-7-7 Section EII 5.8 and Methods of Soil Analysis, Part 1 and Part 2. PNL-MA-567 is distributed and controlled by PNL Document Control. Project staff shall maintain in a Laboratory Record Book (LRB) an accurate record of what was done and the results obtained. Any departures from planned methodology shall be documented and justified in the LRB after approval from the Project Manager or Task Leader as stated in Section 15.2 of this QAPjP.

The Project Manager shall maintain a list of LRB's and the LRB custodian.

Laboratory Record Books shall be maintained in accordance with additional requirements in PNL-MA-70 Administrative Procedure PAP-70-1701.

New technical Procedures, whether they will be included in PNL-MA-567 or not, must be developed in accordance with PNL-MA-70 Administrative Procedure **PAP-70-1101, Test Planning, Performance, and Evaluation** and controlled in accordance with Administrative Procedure **PAP-70-601, Document Control.** (All technical Procedures shall be distributed and controlled by PNL Document Control.) A planned technical procedure change that requires or causes a change in precision, accuracy, completeness, comparability or representativeness (PARCC) shall be reported to the client, whereupon the client, with input from the Project manager, will decide on how to proceed.

A planned technical procedure change that does not constitute a change in any of the PARCC shall be documented in the Laboratory Record Book.

Unplanned deviations from technical procedures are addressed in Section 15.2 of this QAPjP.

All technical procedure changes will be addressed in the final report to WHC.

22.0 DOCUMENT REVIEWS

Document reviews of reports to the client shall be performed in accordance with the requirements in Administrative Procedure, **PAP-70-604, Independent Technical Review.**

23.0 REFERENCES

ASTM D 421-85. Standard Practice for Dry Preparation of soil Samples for Particle-Size Analysis and Determination of Soil Constants. Annual Book of ASTM Standards V 4.08.

ASTM D 422-63. Standard Test Method for Particle-Size Analysis of Soils. Annual Book of ASTM Standards. V 4.08.

ASTM D 854-83. Standard Test Method for Specific Gravity of Soils. Annual Book of ASTM Standards. V 4.08.

ASTM D 4129-88. Standard Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection. Annual Book of ASTM Standards. Vol. 11.01.

Electron Microscopy of Soils and Sediments: Techniques. Smart, P., and Tovey, N.K., Clarendon Press, Oxford. 1982

Environmental Science and Technology. V 23, Testing Readsorption of Trace Elements during Partial Chemical Extractions of Bottom Sediments. Belzile, N., Lecomte, P., and Tessier, A. p 1015-1020. 1989.

Fuel, V 62, Scheme for Density Separation and Identification of Compound Forms in Size-fractionated Fly Ash. Mattigod, S. V., and Ervin, J. O. p 927-931, 1983.

Methods of Soil Analysis, Part 1. Physical and Mineralogical Methods, Chapter 8, Petrographic Microscope Techniques. Cady, J. G., Wilding, L. P., and Drees, L. R. p 185-218. American society of Agronomy-Soil Science Society of America. Madison, Wisconsin. 1986.

Methods of Soil Analysis, Part 1. Physical and Mineralogical Methods, Chapter 12, X-ray Diffraction Techniques. Whittig, L.D., and Allardice, W. R. p 331-362. American society of Agronomy-Soil Science Society of America. Madison, Wisconsin. 1986.

Methods of Soil Analysis, Part 1. Physical and Mineralogical Methods, Chapter 21, Water Content. Gardner, W. H. p 493-544. American society of Agronomy-Soil Science Society of America. Madison, Wisconsin. 1986.

Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, Chapter 9, Exchangeable Cations. Thomas, G. W. p 159-165. American society of Agronomy-Soil Science Society of America. Madison, Wisconsin. 1986.

APPLICABLE ADMINISTRATIVE PROCEDURES:

Administrative procedures applicable to the 100 Area Soil Washing Bench-Scale Test Procedure are listed below. Other Administrative Procedures can be found in the PNL-MA-70 manual but are not applicable to this work at this time. If the current scope of work changes, those procedures not currently identified as applicable shall be reevaluated for applicability. PAPs may be found in Volume II of PNL-MA-70, Quality Assurance Program.

PAP-70-201	INDOCTRINATION AND TRAINING
PAP-70-205	QUALITY ASSURANCE PLANS
PAP-70-208	IMPACT LEVELS
PAP-70-301	HAND CALCULATIONS, GENERAL
PAP-70-401	PREPARATION, REVIEW, AND APPROVAL OF PURCHASE REQUISITIONS
PAP-70-404	OBTAINING SERVICES VIA WORK ORDERS
PAP-70-601	DOCUMENT CONTROL
PAP-70-602	DOCUMENT CHANGE CONTROL
PAP-70-604	INDEPENDENT TECHNICAL REVIEW
PAP-70-801	MATERIAL IDENTIFICATION AND CONTROL (TESTING AND EXPERIMENTATION)
PAP-70-1101	TEST/ANALYSIS PLANNING, PERFORMANCE AND EVALUATION
PAP-70-1201	CALIBRATION CONTROL SYSTEM
PAP-70-1301	HANDLING, STORAGE AND SHIPPING
PAP-70-1501	NONCONFORMANCE REPORTS
PAP-70-1502	CONTROLLING DEVIATIONS FROM QA REQUIREMENTS AND ESTABLISHED PROCEDURES
PAP-70-1602	CORRECTIVE ACTION
PAP-70-1701	RECORDS SYSTEM
SCP-70-314	SOFTWARE CONFIGURATION MANAGEMENT
SCP-70-315	CONVERSION TESTING, VERIFICATION, AND/OR VALIDATION OF SOFTWARE
SCP-70-316	SOFTWARE APPLICATION CONTROL
SCP-70-317	TRANSFER OF SOFTWARE, DATA AND/OR DOCUMENTATION
SCP-70-318	CONTROL OF DATA BASES

REFERENCES

100 Area Soil Washing Treatability Test Plan, DOE/RL-92-51 Draft, A, November 1992.

100 Area Soil Washing Bench-Scale Test Procedures, Rev. 0, November 1992.

EPA QAMS-005/80, Appendix A, EPA 60/4-83-004, February 1983.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update. Method 1311.

References for procedures listed in Table 6.3 will be added by a revision to the References Section of this QAPjP as soon as the necessary clearance is obtained for the manual where these procedures are located.

The Analytical Procedures from Table 6.1 are listed by number and title in Table 6.3 of this QAPjP.

PNL-MA-567, Procedures for Groundwater Investigations, Site Characterization and Assessment Section, "AD-4, Sediment Sample Chain-of-Custody," and "DM-1 Analytical Data Handling and Verification."

Appendix C

Health and Safety Plan

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CONTENTS

FIGURE

1.0 INTRODUCTION

1.1 GENERAL CONSIDERATIONS AND REQUIREMENTS

The purpose of this Health and Safety Plan (HSP) is to establish standard health and safety procedures for Pacific Northwest Laboratory (PNL) employees engaged in performing laboratory bench scale soil washing tests. These laboratory tests include wet sieving contaminated sediments, bench top sediment attrition tests, chemical extraction tests, column heap leaching tests and waste water treatment tests as described in the accompanying Bench Scale Test Procedures.

All PNL staff performing these tests shall do the following.

- Read the HSP and attend a kick-off safety meeting to review and discuss the HSP.
- Follow all relevant health and safety procedures in this HSP, the Environment, Safety and Health Plan and the Chemical Management and Hygiene Plan both issued by the Earth and Environmental Sciences Center and all Radiation Work Procedures posted outside the doors of the relevant 3720 building laboratories to be used on this project.

Employees are encouraged to bring any questions and concerns to the project manager. If a pertinent issue arises the project manager will determine the need to change and specify changes in the referenced documents.

1.2 DESIGNATED SAFETY PERSONNEL

Each laboratory in 3720 building has a designated laboratory monitor whose name is posted on the wall outside the door. This person is responsible for worker's safety and health within the day to day operations. Conversely, no one will perform work within a laboratory without verbal approval of the laboratory monitor.

1.3 TRAINING

All staff performing laboratory work within the designated labs in 3720 will be radiation worker trained personnel. Further, before engaging in any laboratory work all PNL staff must fill out a training questionnaire which is used to determine which PNL and PNL Center-specific course must be taken. The need for refresher training is automatically tracked once a worker has completed the initial training. Many of the training courses must be completed prior to setting foot in the laboratory. For the bench top soil washing tests to be performed in this project the following courses are mandatory before entering the laboratory setting or alternatively attendance is required at the next course offering after the need to work in the labs is established. Courses relevant to the bench top soil washing include:

1. Environmental Safety and Health OJT
2. Emergency Procedures for 3720
3. Use of Portable Fire Extinguishers
4. Safety Shower/Eyewash Use
5. Radioactive Material Packaging
6. Hazardous and Mixed Waste Management
7. Environmental Safety and Health - OJT Mentoring Program
8. Environmental Safety and Health - OJT Chemical Hygiene
9. Hazard Communication Staff Laboratory Worker
10. Environmental Safety and Health OJT Radiation Worker
11. Radiation Safety
12. Laboratory Hood Safety

Again, the laboratory monitor for each lab also instructs each worker about specific requirements and operations in each particular lab. Finally, one person in each technical group must be a certified Hazardous and Radioactive Material Shipping Representative. For the bench-scale soil washing testing V. L. LeGore (3720 Room 303) is the certified shipping representative who will receive all samples and ship all samples to the analytical labs and WHC, when applicable.

1.4 RADIATION DOSIMETRY

All personnel and visitors to the radiation zone laboratories must be assigned a PNL 5-chip multidosimeter. Visitors must be escorted by a qualified PNL Radiation Protection Technician (RPT) and qualified member of the project technical staff during visits inside the labs. The visitors will be briefed on the requirements set forth in the Radiation Work Permits for each lab prior to entry.

2.0 GENERAL PROCEDURES

The following personal hygiene and work practice guidelines are intended to prevent injuries and adverse health effects. A radiation and chemical laboratory poses a multitude of health and safety concerns because of the variety and number of hazardous substances present. These guidelines represent the minimum standard procedures for reducing potential risks associated with this project and are to be followed by all laboratory workers at all times.

2.1 GENERAL WORK SAFETY PRACTICES

2.1.1 Work Practices

The following work practices must be observed.

- Eating, drinking, smoking, taking certain medications, chewing gum, and similar actions are prohibited while in the laboratory.
- Personnel shall avoid direct contact with contaminated materials unless necessary for sample manipulation or required observation. Surgeon's gloves and safety glasses are required at all times when performing work on the contaminated soils.
- While operating in the controlled zone, personnel shall use the 'buddy system' where appropriate or be in visual contact with someone outside of the controlled zone.
- Requirements of PNL radiation protection and RWP manuals shall be followed for all work involving radioactive materials or conducted within a radiologically controlled area.
- Do not handle soil, waste samples, or any other potentially contaminated items unless wearing the protective gloves specified in the RWP.
- Be alert to potentially changing exposure conditions as evidenced by such indications as perceptible odors.
- Follow all provisions of each lab-specific RWP.

2.1.2 Personal Protective Equipment

- Personal protective equipment will be selected specifically for the hazards identified in the RWP.

- Levels of protection shall be appropriate to the hazard to avoid either excessive exposure or additional hazards imposed by excessive levels of protection. The RWP contains provisions for adjusting the level of protection as necessary. These personal protective equipment specifications must be followed at all times, as directed by the radiation protection technician (RPT), and project manager.

- Each employee must have available safety glasses, and protective footwear to wear if specified in the RWP.

- Personnel should be alert to the symptoms of fatigue and its effects on the normal caution and judgment of personnel.

2.1.3 Personal Decontamination

- The RWP describes in detail methods of personnel decontamination, including the use of contamination control corridors and step-off pads when appropriate.

- Thoroughly wash hands and face before eating or putting anything in the mouth to avoid hand-to-mouth contamination.

- Prior to each exiting from the radiation control labs personal surveys of body and protective clothing shall be performed as appropriate.

2.1.4 Emergency Preparation

- A multipurpose dry chemical fire extinguisher shall be available in the hall outside every lab. A pressurized spray eye wash unit shall be available in each lab where there is potential for contamination of personnel to an extent warranting such emergency measures.

3.0 SCOPE OF WORK AND POTENTIAL HAZARDS

While the information presented in the 100 Area Soil Washing Treatability Test Plan (DOE/RL-92-51) is believed to be representative of the constituents and quantities of wastes at the time of discharge, the present chemical nature, location, extent, and ultimate fate of these wastes in and around the liquid disposal facilities are not certain. The emphasis of the bench top soil washing studies will be to characterize the nature and extent of contamination in the two trench sediments and collect data to determine the efficacy of performing soil washing to concentrate contaminants in a significantly reduced volume of soil.

3.1 WORK TASKS

Work tasks are described in the Bench Scale Test Plan.

3.2 POTENTIAL HAZARDS

Existing data indicate that hazardous substances may be encountered during laboratory testing; these include radionuclides, heavy metals, and chemical extractant used to leach the soil.

Potential hazards include the following:

- External radiation (gamma and to a lesser extent, alpha and beta) from radioactive materials in the soil
- Internal radiation resulting from radionuclides present in contaminated soil entering the body by ingestion or through open cuts and scratches
- Internal radiation resulting from the inhalation of particulate (dust) contaminated with radioactive materials
- Inhalation or ingestion of particulate (dust) contaminated with inorganic or organic chemicals, and toxic metals

- Dermal exposure to soil or wash water contaminated with radionuclides
- Dermal exposure to soil or wash water contaminated with inorganic or organic chemicals, and toxic metals
- Physical hazards such as noise and electrical shocks from bench top equipment
- Slips, trips, falls, bumps, cuts, pinch points, and other hazards typical when performing wet chemical studies in the laboratory.

3.3 ASSESSMENT AND MITIGATION OF POTENTIAL HAZARDS

The likelihood of significant exposure (100 mR/h or greater) to external radiation is remote and can be readily monitored and controlled by limiting exposure time, increasing distance, and employing shielding as required.

Internal radiation by inhalation or inadvertent ingestion of contaminated dust is a minor concern because most of the work will be performed with wet soil but will be monitored. Appropriate respiratory protection, protective clothing, and decontamination procedures will be implemented as necessary to reduce potential inhalation, ingestion, and dermal exposure to acceptable levels.

Exposure to toxic chemical substances through dermal exposure is not expected to pose a significant problem for the identified tasks given the use of the designated protective clothing. The appropriate level of personal protective clothing and respiratory protection are specified in the RWP's.

Chemical exposure by inhalation of contaminated dust is not expected to pose a significant hazard because of the relatively low concentrations of chemicals in soil and low concentration of dust in the ambient air.

4.0 ENVIRONMENTAL AND PERSONAL MONITORING

All laboratories to be used in the bench top studies have continuous air monitoring devices and radiation detection devices for self monitoring. The self monitoring instruments must be used prior to each person's exiting the lab.

4.1 AIRBORNE RADIOACTIVE AND RADIATION MONITORING

The building radiation protection technician monitors airborne radioactive contamination levels and external radiation levels. Action levels will be consistent with derived air concentrations and applicable guidelines.

Appropriate respiratory protection shall be required if conditions are such that the airborne contamination levels may exceed an 8-h derived air concentration (e.g., the presence of high levels of uncontained, loose contamination on exposed surfaces or operations that may raise excessive levels of dust contaminated with airborne radioactive materials, such as during soil homogenization).

If, in the judgement of the radiation protection technician, any of these conditions arise, work shall cease until appropriate respiratory protection is provided.

5.0 PERSONAL PROTECTIVE EQUIPMENT

The level of personal protective equipment required is specified in the RWP for each laboratory. Personal protective clothing and respiratory protection shall be selected to limit exposure to anticipated chemical and radiological hazards. Work practices and engineering controls as described in the RWP will also be used to control exposure, because a personal protective equipment ensemble alone cannot protect against all hazards. The following guidelines will be used to specify personal protective equipment ensembles, based on the potential hazards determined in the RWP:

- Occupational Safety and Health Standards, 29 CFR 1910.120 (OSHA 1988a)
- Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities (NIOSH et al. 1985).

6.0 DECONTAMINATION PROCEDURES

Bench top soil washing studies require the use of soils with known chemical and radiological contamination. Consequently it is possible that personnel and equipment could be contaminated with hazardous chemical and radiological substances.

Specific decontamination procedures are provided in the RWP and assistance from the building RPT.

6.1 PERSONNEL DECONTAMINATION

All personnel who exit the radiation zones in the laboratory will first self monitor and then use the hand and shoe counter located in the hallway before proceeding into the common hallways in the 3720 building. In the event that contamination of protective clothing, the lab worker, or the laboratory itself is discovered the RPT shall be notified. Protective clothing that can be cleaned will be removed, bagged, and sent to the laundry. All wash liquids used for decontamination purposes must be properly disposed of in accordance with applicable PNL, state and federal regulations.

At the RPT's discretion, nasal smears may be taken for counting and analysis. Health Physics Dosimetry shall also be notified, and the determination for further bioassay, if needed, will be made at that time. Lab-specific radiation decontamination procedures are provided in the RWP or as specified by the onsite RPT.

6.2 EQUIPMENT DECONTAMINATION

Equipment decontamination methods will generally consist of chemical washing with a detergent and water or other decontamination solution. Rinsing with a dilute nitric acid solution may be necessary to remove metal oxides and hydroxides. Wash liquids used for decontamination purposes must be properly disposed of in accordance with applicable PNL, state and federal regulations.

Equipment radiologically contaminated beyond useful limits will be disposed of as radioactive solid waste at the end of the project. When appropriate, disposable sampling equipment will be used to eliminate the need for decontamination.

6.3 MONITORING EQUIPMENT

All possible measures should be taken by personnel to prevent or limit the contamination of any monitoring equipment used. In general, air monitoring instruments will not be contaminated by chemicals unless splashed or set down on contaminated areas. Any delicate instrument that cannot be easily decontaminated should be protected while it is being used by placing it in a bag and using tape to secure the bag around the instrument. Openings in the bag can be made if needed (e.g., for sample intake, exhaust, electrical connections).

7.0 CONTINGENCY AND EMERGENCY RESPONSE PLANS

The following procedures have been established to deal with emergency situations that might occur during laboratory operations. As a general rule, in the event of an unanticipated, potentially hazardous situation indicated by instrument readings, visible contamination, unusual or excessive odors, or other indications, lab workers shall temporarily cease operations and call the RPT. Any individual leaving a radiologically controlled area needs to be released by a radiation protection technician, even if that individual is going to the first aid station or the hospital. If this cannot be accomplished, for whatever reason, the RPT must accompany the individual to the first aid station or the hospital.

7.1 EMERGENCY SIGNALS

Signal	Meaning	Action
Gong (2 Strokes per second)	Fire	Evacuate building. Move upwind. Keep driveways clear
Siren, steady blasts, 3 to 5 minutes	Area evacuation	Proceed promptly to designated stages area. Listen for emergency information. Follow instructions.
Crash alarm telephone bell, steady ringing, Room 112	Area or plant emergency	Answer crash alarm telephone Relay message exactly as received to the Building emergency Director.
Wavering Siren	Take cover	Stay inside, await instructions
Constant Air Monitor High-room air activity		Leave room, contact Radiation Protection (376-3083) and/or Building Manager (376-0147).

Note: Recorded message and sound of signals on 373-2345.

When leaving the building meet at the staging area shown in Figures 1 and 2.

3720 Building Evacuation Plan

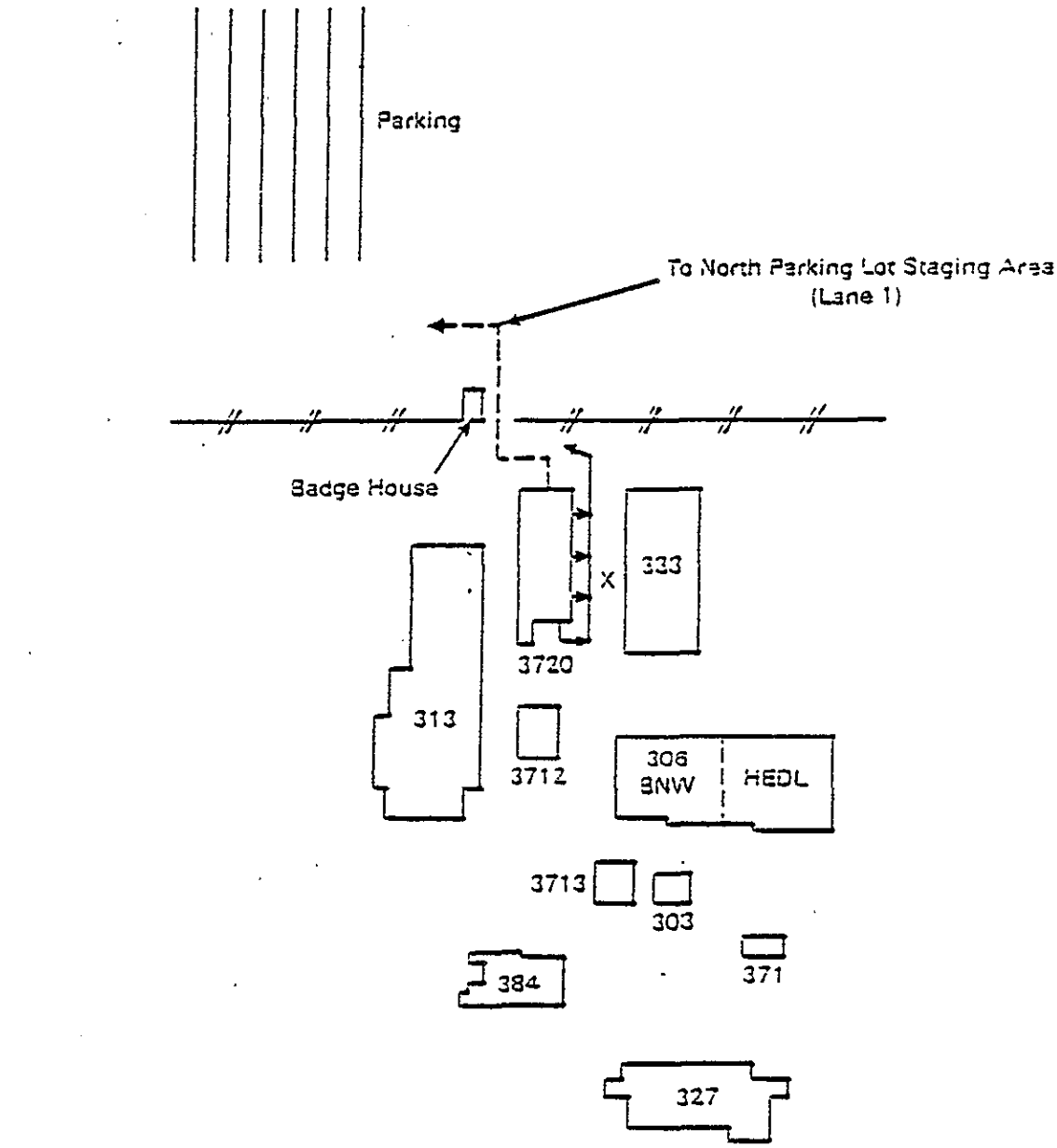

FIGURE 1. Staging Area in North Parking Lot

7.2 PROCEDURE FOR PERSONNEL INJURED IN THE RADIATION ZONE

If an injury occurs, fellow laboratory members will provide appropriate assistance. Only trained, certified personnel should attempt to give first aid, phone 375-2400 and ask for immediate assistance. If able, the injured person should proceed through decontamination to the nearest available source of first aid which is in the 3706 Building (376-3315).

On notification of a serious injury in the radiation zone, call the single point of contact emergency phone number 375-2400 and/or Emergency Medical Aid Station 811-0000.

7.3 PROCEDURES FOR FIRE AND EXPLOSIONS

The dry chemical fire extinguishers that are required outside all radiation zone laboratories are effective for fires involving ordinary combustibles (e.g., wood, plastics), flammable liquids, and electrical equipment. They are appropriate for small, localized fires such as a garbage can of waste, a small burning piece of laboratory equipment, or a hood fire. No attempt should be made to use the provided extinguishers for well-established fires or large areas or volumes of flammable liquids. Call the emergency phone number 375-2400.

In the event of a fire or explosion, the following steps are to be taken.

1. Actuate fire alarm pull box. (The building fire alarm system is tied directly to the 300 Area Fire Station.)

2. Telephone the PNL Single Point Contact on 9-375-2400 to report the fire.

3. Fire extinguishers, strategically located throughout the building, may be used to control small fires; however, NO personal risk is to be taken for this purpose.

   Always actuate a fire alarm pull box before attempting to control even a small fire. Report the fire to 9-375-2400, or have a coworker make the call.

4. If a fire alarm is actuated, all personnel should leave the building and walk upwind at least 100 feet from the building.

5. If you actuate a fire alarm pull box, ensure someone goes to the main entrance to the building to meet the Fire Department personnel with details as to location, hazards, and any specila recommendations.

6. After the fire has been extinguished --

   All equipment used and clothing worn by firemen and other personnel assisting them must be checked for contamination by Radiation Protection personnel.

   If the site of the fire is restricted, the area should not be disturbed until the investigations have been completed.

7. If a fire extinguisher has been discharged, notify your Building Manager. The Building Manager will contact PNL Safety to arrange for the extinguisher to be refilled.

If the fire cannot be readily controlled, take the following steps.

1. On discovering a fire or explosion in the lab alert all staff to evacuate and call the emergency number 375-2400.

2, Isolate the fire to prevent spreading if possible.

3. Clear the area of all personnel working in the immediate vicinity.

7.4 PROCEDURE FOR FAILURE OF MONITORING EQUIPMENT

If laboratory monitoring equipment fails to operate properly, the RPT shall be notified and they will then determine the effect of the failure on continuing operations. If the failure may compromise health and safety procedures or jeopardize the safety of personnel, all personnel shall leave the radiation zone until the equipment is repaired or replaced.

7.5 EMERGENCY TELEPHONE NUMBERS

It is preferred that you use the PNL single point of contact emergency response telephone number 375-2400 and allow them to respond accordingly and alert others. If per chance something goes wrong, these other numbers may prove useful.

Local resources:	Hanford Emergency Response Team	373-3800
Medical Aid Station	3706 Building	376-3315
Emergency Medical Aid	300 Area	811-0000
Ambulance:	Hanford Fire Department (they will dispatch ambulance)	373-3800
Hospital:	Kadlec Medical Center, Richland	946-4611
Police (local or state):	Hanford Patrol Operations Center	373-3800
	300 Area Operations	376-3505
Fire department:	Hanford Fire Department (300 Area)	376-3301
Poison Control Center:		800-572-5842
Radiation Protection:	PNL	376-4703

EMERGENCY CONTACTS

Industrial Safety:	P. A. Wright (PNL)	376-1634
Radiation Protection:	T. Moreno (PNL)	376-3083
Health Physics:	J. R. Berry (PNL)	376-3057
Technical Lead:	R. J. Serne (PNL)	376-8429
Environmental Reporting:	W. J. Bjorklund (PNL)	373-1969

8.0 REFERENCES

PNL-MA-6 Radiation Protection

PNL-MA-43 Industrial Hygiene, Occupational Safety and Fire Protection

PNL-MA-11 Emergency Preparedness

Occupation Safety and Health Standards 29 CFP 1910.120 (OSHA 1988a)

Occupation Safety and Health Guidance Manual for Hazardous Waste Site Activities (NIOSH et al. 1985)

DOE. 1992. 100 Area Soil Washing Treatability Test Plan, DOE/RL-92-51 Decisional Draft, September 1992, US Department of Energy, Richland, Washington.

APPENDICES

Appendix A Earth and Environmental Sciences Center Environment, Safety and Health Plan

Appendix B Chemical Management and Hygiene Plan for Earth and Environmental Sciences Center

Appendix C 3720 Building Radiation Work Procedures (RWP's)

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